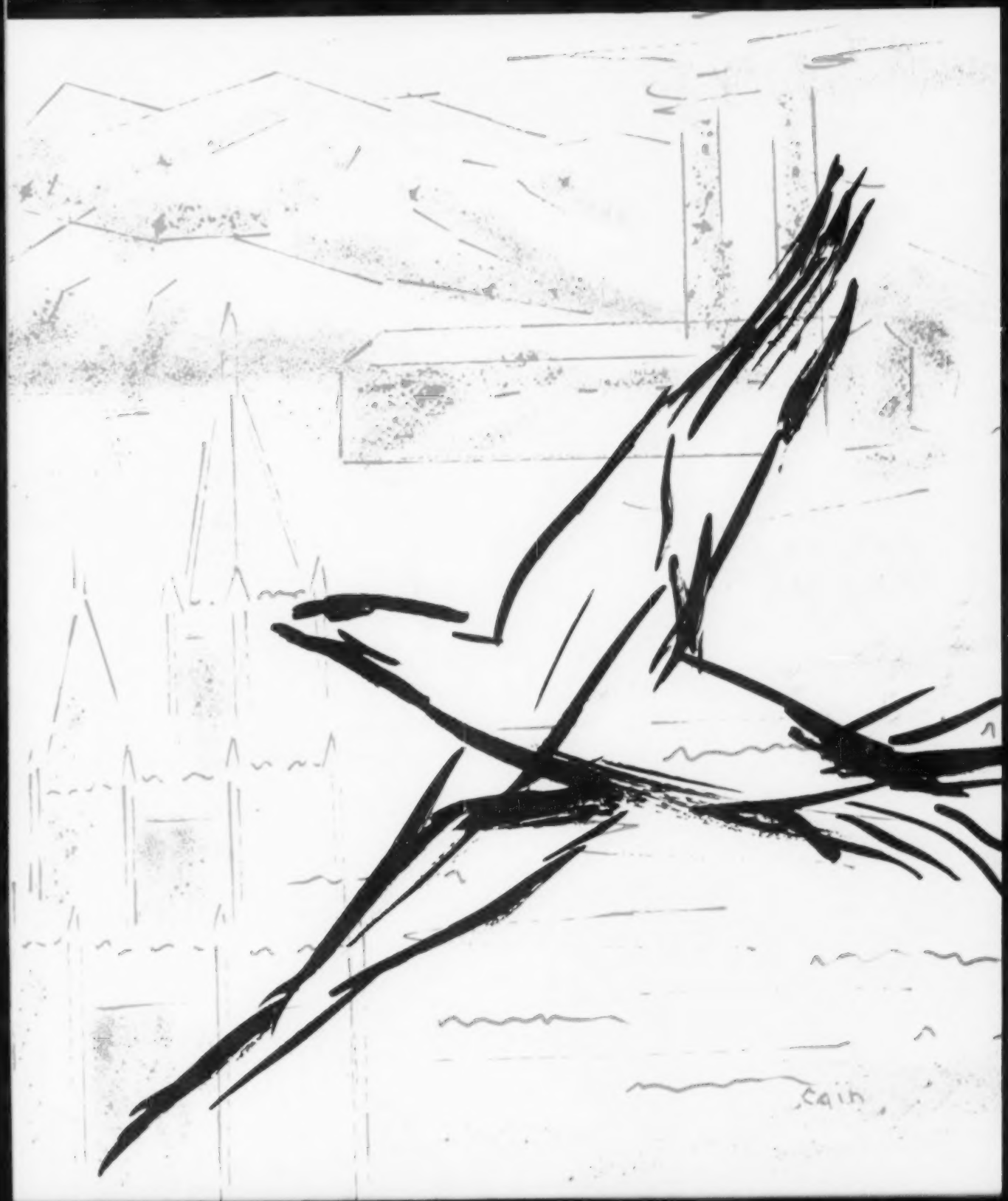
JOURNAL OF

METALS

OCTOBER 1960



One of a series

New Light on a Compound Semiconductor

Pictured is a new and unusual transistor . . . made from a *compound* semiconductor. Its electronic properties are greatly affected by *light*. It is a *field-effect* transistor having input impedances up to 100 megohms (versus 1,000 ohms for junction transistors). Its unique combination of properties has enabled it to perform some novel circuit functions not possible with other transistors.

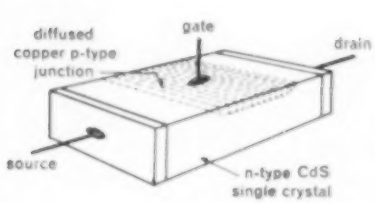
Still in the early experimental stage, this phototransistor is a tangible result of the General Motors Research Laboratories' program on semiconductors — particularly the group II-VI compound, cadmium sulfide. Behind its development lies the steady accumulation of (1) know-how in crystal growing, doping, and contact preparation and (2) information about CdS's intriguing solid state properties (red or green luminescence, high photoconductivity, short relaxation times, etc.).

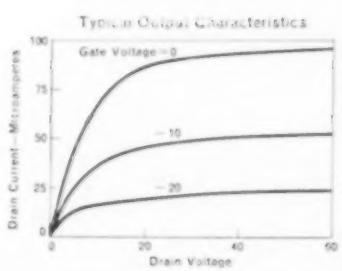
For the researcher, this three-terminal device is adding a new dimension to the fundamental understanding of semiconductors. For instance: GM Research scientists have uncovered the important role of photo-generated holes in modulating the conductance of this intrinsic semiconductor and have determined the hole drift mobility through a new theoretical analysis.

These semiconductor investigations illustrate the dual aim of GM Research: contributions to the science, advances in the technology of important new subject areas. Such research is the initial step in General Motors' continuing quest for "more and better things for more people."

General Motors Research Laboratories

Warren, Michigan





JOURNAL OF

METALS

Vol. 12, No. 10
October, 1960

COVER: Sea gull, temple, lake, and smelter all goes to say, Rocky Mountain Minerals Conference, to which this issue is dedicated. Extractive metallurgists will find a number of timely articles on beryllium and other metals within these covers.

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WESTERN ADVERTISING REPRESENTATIVE: Dillenbeck-Galavan Inc., 266 S. Alexandria Ave., Los Angeles 4, Calif.

Address insertion orders and copy to: Advertising Department, JOURNAL OF METALS, 29 West 39th Street, New York 18, N. Y. Send plates to: JOURNAL OF METALS, C/O Lew A. Cummings Co., Inc., 215 Canal St., Manchester, N. H. Published the first day of each month by the American Institute of Mining, Metallurgical and Petroleum Engineers, Inc., 29 West 39th Street, New York 18, N. Y. Telephone: PEnnsylvania 6-9220. Subscription $10 per year for non-AIME members; $6 for AIME members, or $4 additional for members, only in combination with a subscription to "Mining Engineering" or "Journal of Petroleum Technology." Single copies, $1.50. Transactions of The Metallurgical Society of AIME, $5 additional to members, $20 to non-members, and $25 foreign . . . The AIME is not responsible for any statement made or opinions expressed in its publications . . . Copyright 1960 by the American Institute of Mining, Metallurgical, and Petroleum Engineers, Inc. . . . Registered address, AIME New York . . . Indexed in Engineering Index, Industrial Arts Index, and Chemical Abstracts . . . Second class postage paid at New York, N. Y., and at Manchester, N. H. Numbers of copies in this issue, 12,000.

personnel

• These items are listings of the Engineering Societies Personnel Service, Inc. This Service, which cooperates with the societies of Civil, Electrical, Chemical Mechanical, Mining, Metallurgical, and Petroleum Engineers, is available to all engineers, members and non-members, and is operated on a nonprofit basis. If you are interested in any of these listings, and are not registered, you may apply by letter or resume and mail to the office nearest your place of residence, with the understanding that should you secure a position as a result of these listings you will pay the regular employment fee of 60% of the first month's salary if a non-member, 50% if a member. Also, that you will agree to sign our placement fee arrangement which will be mailed to you immediately after receiving your application. In sending applications be sure to list the key and job number. The New York office of Engineering Societies Personnel Service, Inc., is open for interviews until 7:00 p.m. Thursday evenings.

• When making application for a position, include eight cents in stamps for forwarding application to the employer and for returning, when possible.

—MEN AVAILABLE—

All those listed under "Men Available" have membership in the AIME.

Metallurgist, B.S. Met. Four years R&D on special metals (beryllium, zirconium and uranium), heavy experience vacuum melting. Location open. M-243.

Metallurgist in applied research or product development, B.S. Met. One year product development in cold finished steel plant; 2 years product development in magnetic materials concern. Desire location within 75 mi radius of New York City. M-242.

Research Scientist or Engineer, M.S. Met., B.S. Met., B.S. Physics. Research and development work at university relating to Air Force (v. s.) projects from 1958 to present. One year training in blast furnace operation and practice at large steel corporation. Location, Canada. M-244.

Plant Supervisor, age 52. Over twenty years experience supervision of anodizing and all metal finishing problems, for electrochemical products, automotive parts, bearings, plating equipment and solutions. $10,200. Prefer midwest, northwest. Home: Michigan. Se-1523.

Metallurgical Engineer, M.S. MetE, 35. Ten years combined metallurgical and operating experience in zinc and copper smelters. Speak Spanish. Any Location. Home: Oklahoma. Se-1486.

Metallurgist, Production, Research, Engr. Phys, 37. Eleven years diversified experience in steel industry, research and high temperature alloys including refractory metals. Have very broad engineering and technical background in related fields. Five years responsible supervisory position involving direction of large metallurgical department with staff responsibilities to top management and customer contact on technical subjects. About $12,000. Prefer southwest, south, west. Home: southwest. Se-1412.

Mill Superintendent, Metallurgist, B.S. Ch, M.S. Met, 43. Nine years charge of lead-zinc concentrator, all mill positions including shift boss and assistant mill foreman in copper-zinc mill. Also assaying copper, gold-silver for smelter and refinery. Seven years of miner dressing sales. $9600. Prefer western U.S., foreign. Home: Arizona, Se-1335.

Metallurgist, Superintendent, MetE, 33. Three years charge of casting and wire drawing, shed production, controlled raw materials for aluminum products. One year development, test of new brazing and welding methods for steel components on turbines. Five years research on weldability of newly developed aluminum alloys. $9200. Prefer west, east. Home. Iowa. Se-1690.

Metallurgist or Teacher, MinE, E.M. Met, 57. Twenty years experience; built mills, superintendent, manager for metal mining and milling. Two years sales of fuel additives; chemical manufacturer. One year as associate professor of mining and metallurgy. $6000. Any location. Home: Texas. Se-1772.

Foreman or Superintendent, MetE, 28. Seven years experience in charge of crews, developing undercut blocks on block caving, safety inspection, cut and fill stopes, flotation mill, underground surveying on copper and molybdenum mining. $8500. Prefer western U.S., foreign. Home: Colorado. Se-1648.

Research, Development Engineer, M.E. Phys Met, 39. Seven years research on metals and teaching assistant for university. One year application of heat treatment of metals. $7000. Prefer San Francisco East Bay. Home: San Francisco East Bay. Se-1151.

Chief Metallurgist, MetE, 34. Ten years diversified experience in ferrous and nonferrous foundry, cold and hot forging, production, and research and development titanium, stainless, superalloys, creep, fatigue. $12,000. Prefer west. Home: Connecticut. Se-1025.

—POSITIONS OPEN—

Physical Metallurgist, B.S., M.S., PhD in Metallurgy or Physical chemistry, to develop and apply new and improved materials for sensors for diverse line of instrumentation products. Photo-conductive and photometric measurements; thermoelectric and resistance materials for high temperature measurements. $7000-$10,000 year. Location: New England. W9492.

Foundry Manager, Metallurgical degree, with at least 10 years supervisory production experience including standards and incentives in grey iron jobbing foundry. $10,000-$12,000 year. Location: Massachusetts. W9488(b).

Manager, Process Engineering, graduate Metallurgical Engineer, with interest primarily in metallurgy, but chemical process engineering experience essential. Will be responsible for all phases of chemical and metallurgical plant operations; for the development and successful implementation of standard operating procedures for new and existing processes. Considerable activity in extrusion and forging with future work required in casting, rolling, etc. Salary open, commensurate with experience, training and growth potential. Location: Pennsylvania. W9433.

Metallurgist, young, need not be graduate, for company in precision casting, foundry and heat treating operations. Duties will be primarily to solve production problems involving all alloys. $6000-$7800 year. Company will negotiate fee. Location, New York City. W9397.

Blast Furnace Operator for an integrated steel plant. Will be in complete charge of operations. Must be capable of training native help in supervision and operation. Prefer mature individual. Living quarters available, but schooling is inadequate. At least a two year contract. Apply by letter including complete resume and salary requirements. Excellent climate. Location: South America. F9419.

Metallurgist, Metallurgical Engineer or Chemical Engineer, who has had 5-6 years metallurgical experience in the brass industry. Should have had melting room experience. $7000-$9000 year depending on applicant. Location: western Pennsylvania. W9220.

Address replies for positions to the nearest local office of the Engineering Societies Personnel Service, Inc., *not* to the JOURNAL OF METALS. Offices are located at 29 E. Madison St., Chicago 1, Ill. . . . 57 Post St., San Francisco, Calif. . . . and 8 W. 40th St., New York 18, N. Y.

General Mechanical Foreman with 4-5 years experience in metal refining plant. $10,000 year. Location: Maryland. W8974(c).

Metallurgist, graduate, young, for research and development programs on new alloys. Should have 3-5 years experience in physical metallurgy development work covering a fairly wide variety of ferrous or non-ferrous materials. Experience in large organization with broad range of assignments desirable. Salary open. Location: East. W8973.

Manager, graduate mechanical or metallurgical, with knowledge of alloys helpful, to manage small concern extruding phosphor copper alloys. Should be well versed in extrusion presses, wire drawing, billet casting; knowledge of copper alloys. $10,000 year. Location: Westchester County, N.Y. W8911.

Metallurgist, graduate, service engineering contacting aluminum die casting companies assisting in solving problems in manufacturing. Considerable traveling within 500 mi radius of Chicago, but home every week. Car furnished. $7200-$9000. Employer will pay fee. Location: Chicago area. C8235.

Research Metallurgist and Lecturer, M.S. or PhD, age to 45, to conceive research projects, supervise laboratory experimentation, teach in specialized field, guide graduate research, prepare research proposals and reports for research and education. $8000 up. Location: Rocky Mountain area. C8264.

Consultant Metallurgist, Met, under 50. Wide metallurgical experience in copper, smelting and refining, familiar with operation and engineering in copper producing areas throughout the world. Will provide technical direction for metallurgical plant and design construction, also technical assistance to operate smelters and refineries, and handle development work for new methods. Must be top-notch with sound economic sense. Capable of contact work under variety of circumstances including top management. Salary open, commensurate with experience. Headquarters: Mountain states. Sj-5444-R.

(a) **Junior and Senior Design Engineers,** MinE, C.E., M.E., or MetE degree. Require background in industrial design and drafting with previous experience in mining, milling, smelting or refining plants and facilities desired. Applicants for positions at junior level especially desired, but some seniors also. $7200-$10,200. Headquarters: Utah. Sj-5443-R.

(b) **Construction Engineer,** grad C.E. or M.E. with experience in all phases of plant construction, preferably in mining or mineral industry. Benefits. $7200-$8400. Headquarters: Utah. Sj-5443-R.

Excellent opportunity for B.S./M.S. Metallurgist. Minimum five years' experience; to establish and operate metallographic laboratory in large metropolitan New Jersey research laboratory engaged in semi-conductor and thermoelectric work. Must possess good laboratory technique as well as a sound theoretical background. Salary commensurate with experience. Send resume to Box No. 18JM.

IMMEDIATE OPPORTUNITY FOR

PHYSICAL METALLURGIST

Large metropolitan New Jersey research laboratory currently engaged in semi-conductor and thermoelectric field seeks senior man with broad and proven background in physical metallurgy. Minimum of 5 years' experience; PhD desirable, but not essential. Salary open. Send complete resume to Box No. 17JM.

FIVE-DEEP

Millions in technology

help you profit with UCM's "FIVE-DEEP" Ferroalloys

1 Technology—many million dollars a year, invested in UCM's 600-man research and development center—helps you produce better, more profitable metals. This is one of the 5 intangible but ever-present extra values of Union Carbide Metals' FIVE-DEEP alloys.

It has brought you more than 100 new alloys and metals designed to give you production economies and improved products. Countless millions have been made and saved because of UCM's research, along with these 4 other extra values:

2 Customer Service brings you our integrated experience in the application of ferroalloys to various melting practices. Engineers from 9 UCM field offices travel a million miles a year to provide on-the-scene assistance.

3 Global Ore Sources assure you uninterrupted supplies of ferroalloys. UCM's close association with world-wide mines provides dependable raw material sources.

4 Unmatched Facilities free you from delivery worries. Only UCM gives you 6 plants—3 with their own power facilities—and 17 warehouses, all located for fast shipments by rail, truck, or water.

5 Strictest Quality Control—with over 100,000 tests per month from mines to shipment—makes sure you always get alloys of uniform size and analysis, with minimum fines, lot after lot.

For better metals, production economies, bigger profits, insist on UCM's FIVE-DEEP alloys. Union Carbide Metals Company, Division of Union Carbide Corporation, 270 Park Avenue, New York 17, N. Y., producer of "Electromet" brand metallurgical products.

"Union Carbide" and "Electromet" are registered trade marks of Union Carbide Corporation.

UNION CARBIDE METALS

Only **ELECTROMET** ferroalloys from **UCM** are so deep in extra values to help you.

education

LITERATURE

A new technical data booklet on the consumable electrode method of vacuum melting super alloy steels has been released by Midvale-Heppenstall Co. The new book is written in factual style to supply metallurgists, design engineers, and research and development men with data on vacuum melting, the advantages of metals produced by this method, and the properties of alloys made by the Midvac Process.

Copies of the booklet are available by writing to *Dept. MV, Midvale-Heppenstall Co., Nicetown, Philadelphia, Pa.*

• • •

The 1960 issues of four Russian technical journals, translated into English, will be published during the coming year by the Instrument Society of America, under a grant from the National Science Foundation. Known as the Soviet Instrumentation and Control Translation Series, this is the fourth year of publication. Included in the series are:

Automation and Remote Control (Avtomatika i Telemekhanika), considered to be the leading Soviet journal in the automatic control field. Published monthly by the Institute of Automation and Remote Control of the Academy of Science, U.S.S.R., it carries approximately 150 pages per issue of articles on all phases of automatic control theories and techniques.

Measurement Techniques—(Izmeritel'naia Tekhnika). Approximately 100 pages per issue, published monthly by the Committee of Standards, Measures, and Measuring Instruments of the Council of Ministers, U.S.S.R. Of possible interest to those engaged in the study and application of fundamental measurement.

Instruments and Experimental Techniques—(Pribory i Tekhnika Eksperimenta). Published bimonthly, more than 175 pages per issue, by the Academy of Sciences, U.S.S.R., each issue contains articles relating to function, construction, application, and operation of instruments in various fields of instrumentation.

Industrial Laboratory—(Zavodskaya Laboratoriya). Published monthly by the Ministry of Light Metals, U.S.S.R., it contains approximately 125 pages per issue. Articles appearing are on instrumentation for analytical chemistry, and physical and mechanical methods of material research and testing.

For subscriptions or information, write to Foreign Translations Dept., Instrument Society of America, 313 Sixth Ave., Pittsburgh 22, Pa.

• • •

The American Society for Quality Control is offering *Case Histories on Statistical Methods for Quality Control*, prepared by the society's Metals Technical Committee. The publication is made up of case histories oriented with emphasis on methods rather than on specific areas of application. It is the second in a series.

The general problem which the publication covers is that of increasing open hearth production with existing facilities. The problem was defined by works management and assigned jointly by blast furnace and open hearth supervision. The technical details were reported in the first publication, which was published in Jan. 1958.

Case Histories on Statistical Methods for Quality Control, Series II, (Regression, Correlation and Association) was published by the American Iron and Steel Institute (as was the first publication) in Apr., 1960.

It is available at a cost of $1.00 each from the American Society for Quality Control, 161 West Wisconsin Ave., Milwaukee 3, Wis.

CONTEST

In conjunction with the steel casting industry's centennial observance in 1961, the Steel Founder's Society of America has announced an expanded Product Development Contest.

The purpose of this contest is to encourage the development of steel casting applications through engineering analysis and design. Almost any new development or improvement in the use of steel castings will be eligible in the contest which runs from Oct. 1960, to June 1961.

Prizes totaling $10,000 will be awarded. There will be 22 awards

(Continued on page 817)

METALLURGICAL ENGINEERS . . . . .

Recent Grads For Technical Work In Non-ferrous Extractive Metallurgy

•

Some furnace and process development required

•

Large metals refining plant located in Middlesex County, New Jersey

•

SUBMIT RESUME AND SALARY REQUIREMENTS IN CONFIDENCE:

•

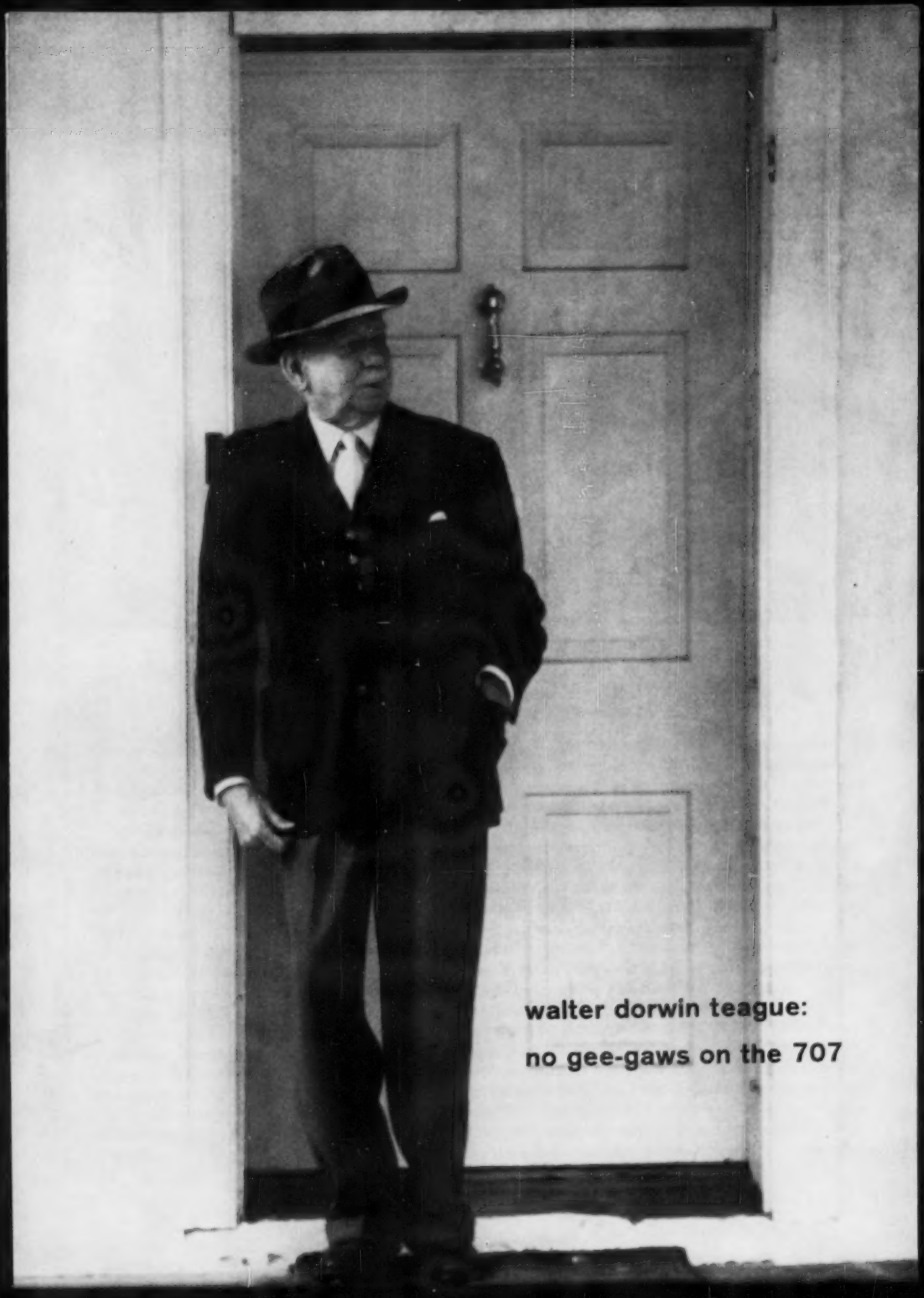
BOX JM 326,
125 West 41 St.,
NYC 36

PHYSICAL METALLURGICAL

For development of high temperature alloys, cermets, and ceramic materials and compatability, stability and physical property studies. Prefer metallurgists, physicists or chemists with graduate studies or research experience in powder metallurgy or ceramics.

Send complete resume in confidence to Employment Manager,

K. H. Soderlund
THE BRUSH BERYLLIUM COMPANY
4301 Perkins Ave.
Cleveland 3, Ohio



walter dorwin teague:
no gee-gaws on the 707

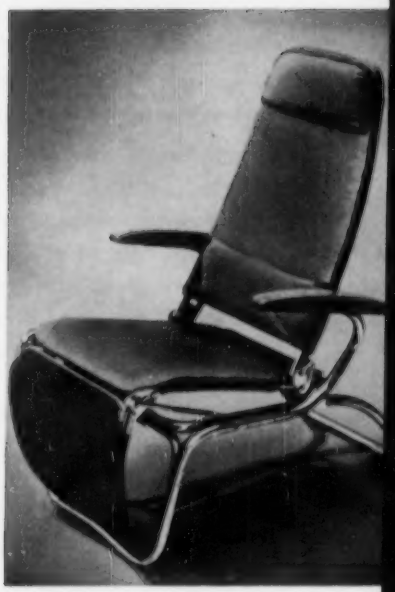
teague talks design

In 1942 a man named Walter Dorwin Teague published "You Can't Ignore Murder," a mystery novel. It is probably the only attempt at bafflement in his long history, because Walter Teague has been making products speak clearly for themselves since the Twenties.

Teague has rightly been called the dean of industrial design. He began as an artist in 1908 designing advertising, books and magazine illustrations. If you're old enough to remember Locomobile and Pierce Arrow ads, Teague designed them. In 1927, Eastman Kodak asked him to redesign their cameras. He made a proposal to spend one week a month in their plant observing production problems and techniques, and *then* redesign the cameras. That simple principle of knowing how a product is made, of understanding all the complexities of the production line, is still a Teague trademark. And Eastman Kodak is still a Teague client.

In the thirty-plus years since that camera assignment, Teague has explored just about every nook and corner of the wonderful world of three dimensions. He designed the classic Marmon 16 automobile in 1930, with his son as collaborator; he was on the Design Board of the 1939 World's Fair. When World War II exploded, the Navy asked Teague to perform design work on their 16-inch guns in order to eliminate possibilities of explosions caused by loading malfunctions. Today he still works for the Bureau of Ordnance. He has 120 people in his organization, a branch in San Juan and task forces scattered across the United States

Teague has designed service stations, railway equipment, plane interiors, heating appliances, business machines and machine tools, offices and furniture, showrooms and department stores, even periodicals. One of the most impressive things he has done is the Boeing 707 jet interiors. Mr. Teague recalls: "Boeing was concerned about their ability to sell the 707 because they hadn't made civilian planes for years. We convinced them to build a full-size mockup of the interior. They gave us *carte blanche* with body engineers to collaborate, and they refused to look at it until it was finished. We made it complete to the tiniest detail. It cost half a million



dollars and helped in selling most of the huge fleet now in the air. Our staff is responsible for the final interiors of the planes purchased by eighteen or more airlines, and for the interiors of executive planes purchased by the United States Government."

Whether mammoth jet airliners or chairs, a product is designed to five principles that are the *modus operandi* of the Teague organization. "First of all, the redesigned product has to work better for our efforts," Teague says. "It should be more convenient to use, more humanized. It must make honest use of the material it's made of, and it's got to be capable of being efficiently manufactured at the proper price. We assiduously avoid gee-gaws or extraneous ornamentation that adds nothing but price to the product. Finally, the design must give the user an emotional pleasure or gratification each time it is used." Selection of the right material for the job is important to each of Teague's requirements. "We endorse no material over another," Teague says. "We first look for the material that is best in keeping with the product's desired personality. Strength and durability are highly important in material selection. Then we make sure our client's tooling can handle the material we have in mind. Above all, it takes the right material to sell the product to the consumer."

Teague has used practically all materials known to man and it is no accident that a great many of his designs use steel in one way or another. "The wonderful thing about steel is its versatility," says Teague. "Its many alloys, old and new, give it adaptability that other materials don't have. Its strength, both in tension and compression, qualifies it for literally thousands of uses. And stainless steel is another reason why steel is a modern metal because it gives so many designs their contemporary look."

The moral is this: steel is an ageless metal, as much at home in the Boeing 707 and today's elegant tableware as it was in medieval days. Its enduring modernity will always be recognized and used by designers like Walter Dorwin Teague.

USS TRADEMARK **United States Steel**

designing with stainless steel

The ideal design has been described as one in which the properties of the material are fully utilized in the finished product. This ideal is difficult to achieve, and no group of materials comes as close to permitting this ideal as stainless steel.

To put it differently, no other material combines so many outstanding properties as the stainless steels. Stainless steels have high tensile strength. All are hardened and made even stronger by cold working. As a family they offer superb resistance to the corrosive effects of an enormous variety of reagents. They have unusual resistance to high temperature oxidation, and are distinguished by relatively low heat conductivity. And on top of their remarkable physical properties, stainless steel's lustrous appearance and sales appeal scarely need mention.

There are over 30 types of stainless steel today.

The history of stainless steel's use is marked by designers who turned a healthy profit with a quality material but mainly with sound ideas. They're the men who used stainless for its strength and sales appeal in toaster covers, its appearance and machinability in wrist watches, its structural properties and cleanability for truck trailer parts. Stainless steel cuts weight and adds years to the life of architectural panels; its corrosion resistance and smooth, pocket-free surface have made it the standard of the dairy industry. There are literally thousands of applications of stainless steel in which designers have utilized one, two or more of the remarkable properties of stainless steel.

By reflecting the color of its surroundings, stainless steel contributes to harmonious design.

Types 302, 410 and 430 make up 75% of stainless steel's uses.

Good design is honest design, whether it uses stainless steel or other materials. Yet the fact remains that there is no other commercial material quite as versatile as stainless steel. The applications shown on these pages are just a few of the hundreds of uses to which stainless is put every day. To learn more about stainless steel and its design properties, call our nearest sales office or write United States Steel, 525 William Penn Place, Pittsburgh 30, Pa.

Successful designers don't substitute stainless steel sections for sections of other materials. They design to stainless' unique properties.

Good design doesn't stop with function and sales appeal. It must also be capable of economical manufacture. As a comprehensive guide, send for the free book described at the right.

Stainless steels represent but a few of the thousands of grades of steel in existence today. United States Steel makes a complete line of stainless steels as well as alloy, high strength and carbon steels. Bring your design problems to us.

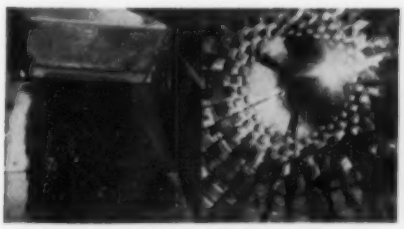
USS is a registered trademark

USS United States Steel

No Material comes close to stainless steel's versatility. By way of illustration, here are a few "opposites" that prove our point.

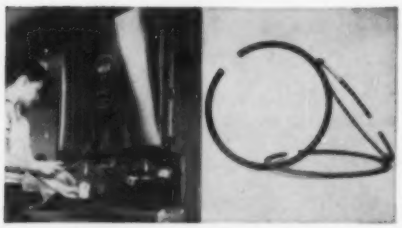
HOT. Temperatures in a jet afterburner reach 1400°F.

COLD. Stainless heat exchanger operates at temperatures as low as —443°F.



WET. Combination of moisture and coal would corrode nearly anything but stainless.

DRY. Rotary dryer is used to dry pharmaceutical ingredients.

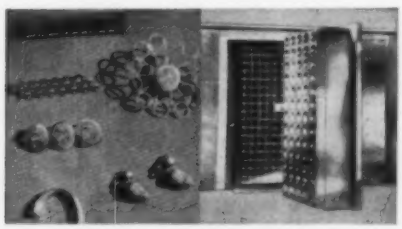


CLEAN. Milk dispensers of stainless steel prevent contamination.

DIRTY. Stainless piston rings resist high temperature corrosion.

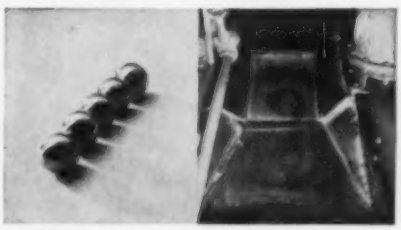
INSIDE. Chemical tanker needs simple wash-out for cleaning.

OUTSIDE. Stainless automobile trim stays bright and good-looking.



LIGHT. Stainless steel jewelry has a light, graceful appearance.

HEAVY. Strength and hardness of stainless combine to make safe doors safe.

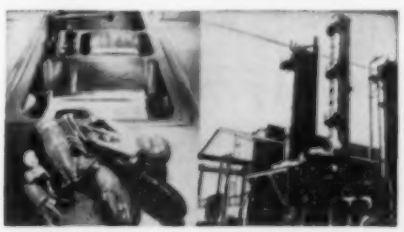


ROLL. Stainless ball bearings take a lot of punishment.

SLIDE. Abrasion resistance of stainless steel makes chutes last longer.

FORM. Stainless steel wrist watch keeps time and beauty.

FUNCTION. Stainless steel muffler defies heat with silence.



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personals

William R. Opie has joined American Metal Climax, Inc. as manager of research at the Carteret, N. J. laboratory of its subsidiary company, Amco Research & Development, Inc. Dr. Opie was previously employed by the Titanium div. of National Lead Co. as research supervisor of the Geochem and Metallurgical Dept.

John Hazel has been promoted to assistant director of research for Republic Steel Corp. Mr. Hazel was formerly the company's chief ceramic engineer and head of the Independence, Ohio Research Center.

Sir Charles Goodeve has been unanimously proposed for nomination as president elect of the British Iron and Steel Institute, by the Institute's Council. Sir Charles will succeed **W. F. Cartwright** as president at the Annual General Meeting in May 1961, and will thus be in office at the time of the Institute's proposed Special Meeting in the U.S. in Oct. 1961. Sir Charles became an honorary member of the Council of the Iron and Steel Institute in 1945; he was elected a vice president in 1956.

James R. Bachman has been appointed manager of the basic refractories laboratory of the Refractories & Chemicals div. of Kaiser Aluminum & Chemical Corp. Bachman has been at the laboratory since 1957, devoting much of his time to refractory applications in the steel industry.

Charles A. Divine, Jr., has been promoted to assistant metallurgist—stainless steels. He was formerly in the company's technical service department.

Daniel W. Machon has been appointed manager of the Santiago, Chile div., of Cerro de Pasco Corp. Mr. Machon, who was until recently in charge of administration for Freeport Sulphur Co. in Cuba, will have similar responsibilities for Cerro in Chile.

Henry S. Rubenstein has been appointed project supervisor, Process Metallurgy, by The Beryllium Corp. of Reading, Pa. He was formerly head of the Development Dept., Bridgeport Brass Co.

Roy W. Drier, professor of theoretical and applied x-rays at Michigan College of Mining and Technology, retired from the faculty on July 11. Dr. Drier's retirement ends a 42-year career, including four years of military duty and five years in industry.

George A. Allison has been promoted to general superintendent of the Electrolytic div. of American Zinc, Lead and Smelting Co., at Monsanto, Ill. Mr. Allison was formerly assistant superintendent of the company's Dumas, Tex., div.

George Havas has been appointed vice president and director of engineering of Kaiser Industries Corp. He has been associated with the company since 1928.

Edward L. Harmon, Jr. is now senior research engineer of the Jet Propulsion Laboratory, California Institute of Technology. He was previously a research metallurgist with Union Carbide Metals Co.

Robert B. Mears, has been appointed assistant vice president in charge of applied research of U.S. Steel Corp., Pittsburgh. Dr. Mears was formerly director of the company's Applied Research Laboratory in Monroeville, Pa.

John C. Douglas has been appointed vice president of operations of Union Carbide Metals Co. He has been with the company since 1934. Mr. Douglas will be succeeded as vice president, technolgy, by **David Swan**, formerly manager, planning. He has been with Union Carbide since 1946.

(Continued on page 766)

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PERSONALS

(Continued from page 764)

Gabriel Husson, head of the steelmaking department of the French Iron and Steel Research Institute (IRSID) has retired. He is succeeded by **Bernard Trentini,** formerly his assistant. Mr. Husson, who has been head of the steelmaking department since the Institute was begun in 1947, will remain active with the organization in a consulting capacity.

Bruce Chalmers, professor of metallurgy at Harvard University, has been awarded the Albert Sauver Achievement Award, sponsored by the American Society for Metals.

Thomas L. Carey has been named director of exploration and mining development for the Armour Agricultural Chemical Co., Atlanta, Ga. He has been mining consultant in the Chicago office of the company since Sept. 1959.

Stephen D. Bechtel, president of the Bechtel Corp., has been selected as the 1961 recipient of the John Fritz Medal, which is awarded annually for notable scientific or industrial achievement in the engineering profession. The award, established in 1902, is sponsored by ASCE, AIME, ASME, AIEE, and AICE. Mr. Bechtel has been associated with the company which bears his name for 42 years.

D. E. Stingel has been appointed works manager of Union Carbide Metals Co. He has been vice president of Carbide Powder Co., and production manager of Union Carbide Olefins Co., since 1958.

John E. Jacobs has been elected vice president, steel operations, of Bethlehem Steel Co.

Julian Glasser and **William E. Few,** formerly associated as consultants in chemical and metallurgical activities, are now associated as president and vice president, respectively, in a new corporation, Chemical and Metallurgical Research, Inc., Chattanooga, Tenn.

W. V. Ward has joined International Nickel Co., as technical representative in their Los Angeles office. He was formerly with Lockheed Aircraft Co.

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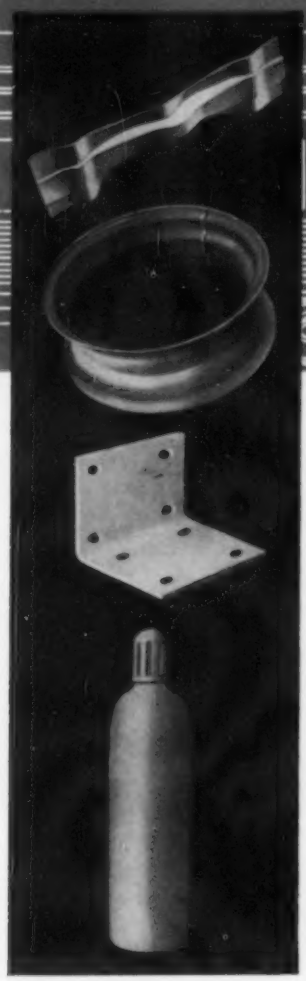

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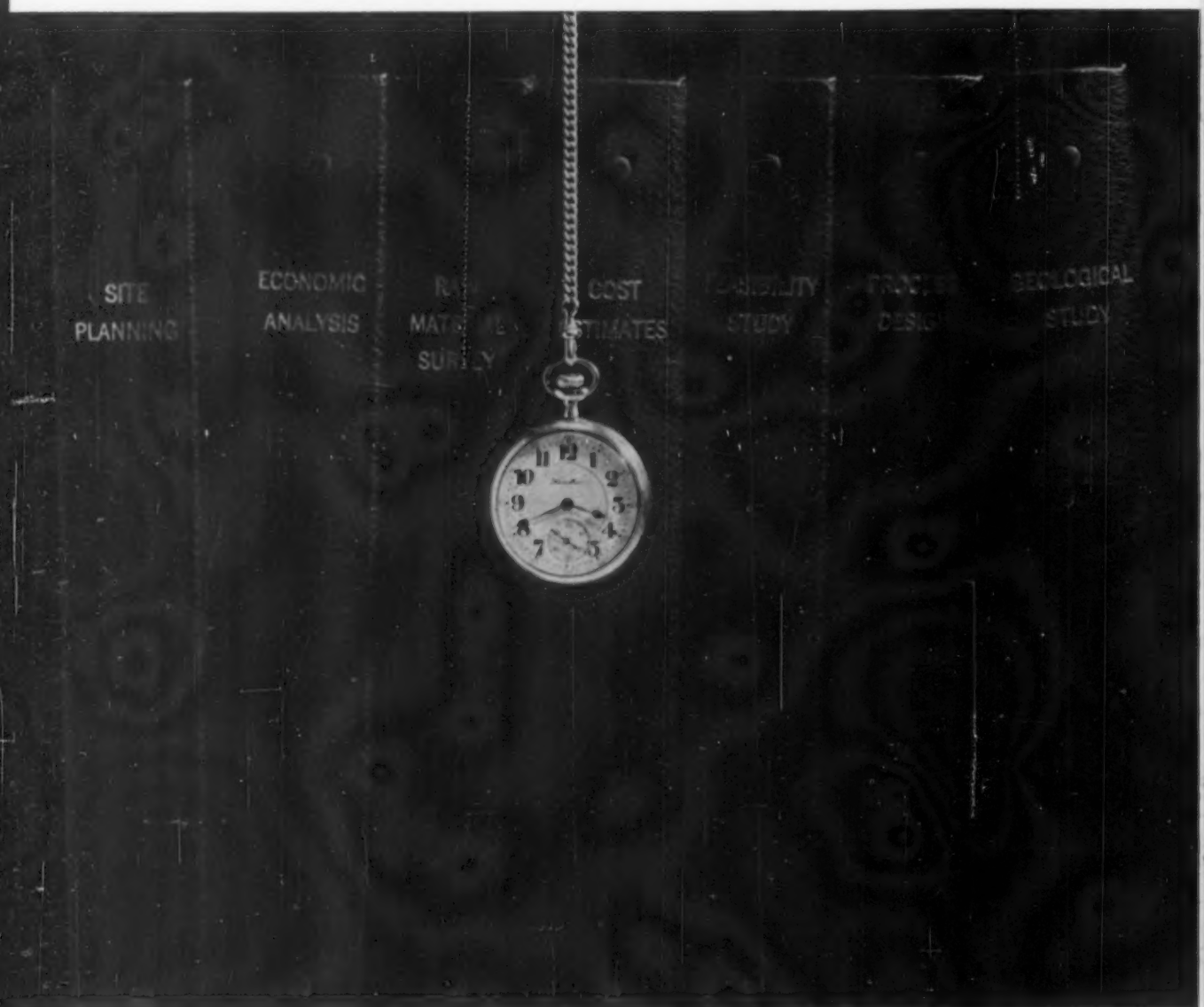

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Forgeable U-Mo ingots

Stauffer-Temescal Co., has markedly reduced grain size in cast tungsten and molybdenum ingot, thus substantially increasing the ductility and workability of these metals without impairing their strength characteristics. This was accomplished by using a combination of electron-beam processing and critical composition control. The same techniques have been successfully applied to the company's range of other ST(electron-beam processed) alloys, including those of columbium and tantalum-tungsten.

The high degree of purification and grain boundary refinement obtained by electron-beam processing has previously produced tantalum-tungsten and columbium alloys sufficiently ductile to be worked in conventional steel fabricating equipment. However, owing to the large crystal size, ingots of pure molybdenum and tungsten still proved difficult to work and extremely brittle at room temperatures. The new modified tungsten and molybdenum ingots are readily workable in conventional equipment with a minimum of special treatment, and the finished products show considerable increased shock resistance.

New Re recovery process

Kennecott Copper Corp. has developed an improved and simplified process for the recovery of extremely high purity rhenium compounds.

During the concentration step, the impurities contained in solution are selectively removed to produce a concentrated solution of high purity rhenium. The purified rhenium solutions may be processed further for the recovery of either high purity rhenium compounds or rhenium metal.

New Norton refractory

Norton Co., has developed *Crystolon 63* a high temperature refractory product which is expected to have a wide application as a relatively low cost structural ceramic. It is said to be high in physical strength and resistance to attack by heat and molten salt. The product has been used experimentally as a lining in aluminum reduction cells, and test results indicate that wall thickness of the cell linings can be reduced up to 75 pct. Reportedly, substantially less electric power is required to operate the cell.

Crystolon 63 is a silicon carbide refractory bonded with silicon nitride. The material used to bond silicon carbide determines the limit of use of the refractory. Silicon nitride bonds are said to exhibit excellent resistance to attack by fused salts.

He-Cooled furnace built

National Research Corp., has developed the first large helium-cooled vacuum arc furnace for the recovery of titanium scrap. Also suitable for handling other reactive and refractory metals, the furnace provides improved operating safety for melting and casting such metals and combines unique features of skull melting by permanent electrode with vacuum melting by consumable electrode.

The furnace's helium gas cooling system replaces the more conventional and more hazardous water cooling normally employed. Single pours of up to 550 lb have been demonstrated.

Other reactive and refractory metals which appear promising for alloying and casting development in the furnace include columbium, tantalum, molybdenum, and tungsten.

The furnace was developed by N.R.C. under contract for the General Services Administration.

Ta element furnace

A tantalum element vacuum resistance furnace that operates in the 4200°F range and is said to be the largest unit yet constructed for brazing, sintering, and heat treating refractory alloys has recently been delivered to the Martin Co. It will be used by Martin for manufacturing research and production of missile parts, including brazing of high temperature honeycomb sections.

The new furnace utilizes a unique resistance element system that allows for expansion of the tantalum heating strips at elevated temperatures. This system consists of a series of serpentine-like heating elements, corrugated for additional strength and supported by shielded water-cooled copper posts.

No refractories are used inside the furnace chamber. Metal heat shields, attached to the walls in "packs", insulate the chamber and allow high-temperature, low-pressure operation.

New Zi-Ti fabricating plant

Calumet & Hecla, Inc., is planning a new, $1.6 million facility at Inkster, Mich., for fabricating special metals. Zirconium and titanium will be the first metals produced at the new center. The company has been producing products from zirconium, titanium and other special materials such as tantalum, columbium, vanadium, molybdenum, beryllium, and chromium in a laboratory pilot plant for five years.

Initially the new plant will produce tubes with outside diameters up to 2¼ in. and rods with diameters up to 2 in. Later, additional finished forms may be added to the line. Full scale production is expected to begin by Jan. 1, 1961. The company's Wolverine Tube div. will operate the new facility.

REPORTER

REPORTER

Cs-In-Rb research

The US Bureau of Mines has scheduled research programs in rhenium, gallium, indium, cesium, rubidium, and scandium during the 1961 fiscal year, now underway.

The rhenium program will include investigations of new sources of the element and development of techniques for the direct separation of rhenium from molybdenite by leaching.

Gallium research will be directed toward accurately determining density data of pure gallium in the range of 30° to 400°C for use in other basic research on pressure-volume-temperature measurements of natural gas and petroleum compounds.

The extraction and separation of cesium and rubidium from their ores, particularly pollucite, will be studied.

The commercial recovery of scandium from uranium plant solutions will be studied. The scandium production potential of various uranium mills will be investigated. Studies, using a radioactive isotope of scandium will be undertaken to find suitable extractants.

The Bureau will attempt to develop processes for recovering nickel and cobalt from laterite ores.

Oxygen plants slated

Linde Co., will build two oxygen plants, one near the Houston Ship Channel, and the other at Pueblo, Colo., The Houston plant will be built for the Sheffield div., of Armco Steel Corp. Linde will supply the plant with 100 million cu ft per month of oxygen. The plant at Pueblo will supply Colorado Fuel and Iron Corp., with 200 million cu ft of oxygen per month.

Gas alloying steel process

Lee Wilson Engineering Co. Inc., has developed a gas alloying process for varying the chemistry of finished, flat rolled steel.

The company's engineers successfully *opened* a coil of steel and handled it in the open condition during annealing. A nylon cord or corrugated metal tape was inserted between each lamination of the coil, spacing the layers of steel. Hot gases were directed at high velocities through the spaces, heating the coil thoroughly and uniformly in a fraction of the time required for conventional batch annealing.

The company states that it is now economically feasible to add or remove carbon, nitrogen, and other alloying elements that can be gasified in the annealing cycle and change the chemical composition of the steel.

Oxygen converter process

National Steel Corp. will install what are believed to be the world's largest furnaces for making steel by the oxygen converter process at its plant in Detroit.

The company will award contracts for two vessels, each rated to produce 250 tons of steel per heat. After contracts are awarded, completion is expected in 18 months, probably by mid-1962.

The new furnaces, which will have a combined capacity of two million ingot tons of steel per year, will partially replace the plant's existing open hearth capacity, currently rated at 3.7 million ingot tons a year. The company will retain all 17 of its existing open hearth furnaces for operation in periods of increased demand for steel.

The rated capacity of the Detroit plant is expected to be increased to 4.2 million ingot tons per year.

The new converters will be bottle-shaped vessels made of heavy steel plate. They will be 29 ft high with a refractory lining as much as 3 ft thick. They are expected to give more uniform control of quality, and greater elasticity of product specifications.

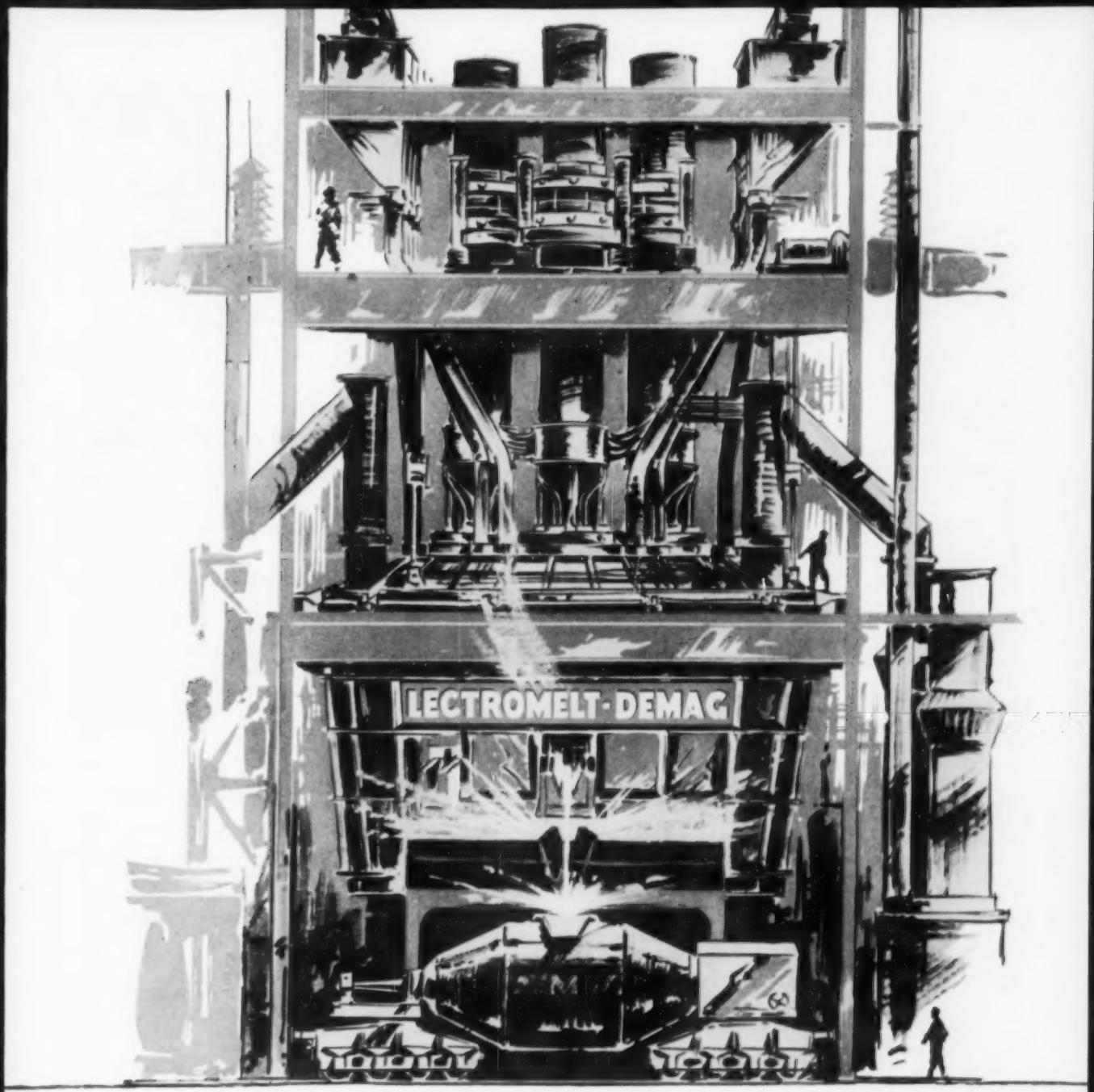
Kaiser refractories record

Kaiser Refractories & Chemicals div. has announced that its new line of tar-bonded refractories has established a new record of 30,625 tons of steel made in 277 heats during a recent campaign. This amounted to an average of 110.5 tons of steel per heat. There was reportedly only a modest comsumption of maintenance material during the campaign.

The company will build a half-million dollar plant at Columbiana, Ohio, for the manufacture of special refractories required in the basic oxygen steel making process. The new plant, to be built next to the company's basic refractories plant, is expected to be in operation by the first of the year. The plant will produce a product similar to that produced at the company's Moss Landing, Calif., facility.

Raw materials for making the special tar-bonded refractories consist of sized deadburned dolomite and periclase, a dense crystalline form of magnesia. Dolomite from the East will be used, and the periclase will be furnished by the Midland, Mich., plant.

Equipment will consist of a specially designed press, rotary heaters, and a mixer where the periclase and dolomite are combined with heated tar and then pressed into brick. After pressing, the brick is cooled and palletized with moisture proof protection.



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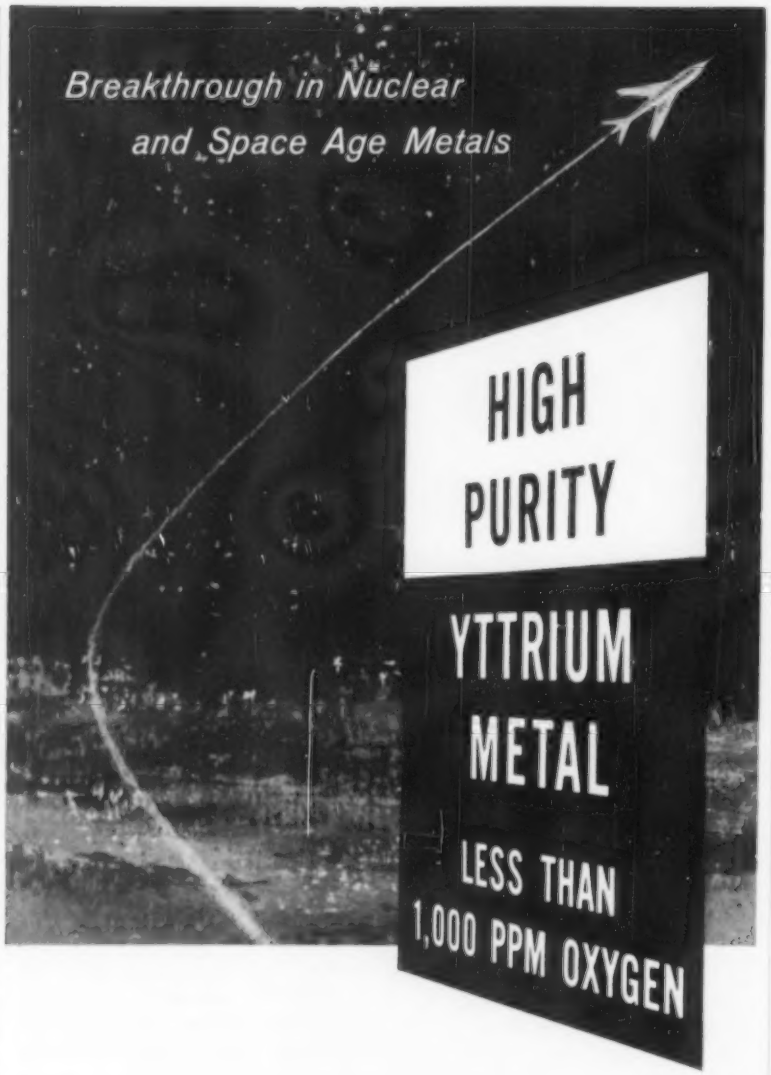

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The Proceedings of a Technical Symposium sponsored by the Nuclear Metallurgy Committee of the Institute of Metals Division, and the Titanium, Uranium, and Uncommon Metals Committee of the Extractive Metallurgy Division, The Metallurgical Society of AIME. San Francisco, February 16-17, 1959.

Edited by W. D. WILKINSON, Argonne National Laboratory

The addition to these Proceedings of an introductory chapter on the metallurgy of plutonium and of an extensive annotated bibliography has made this volume of even greater value to metallurgists working in this particularly important field. Mr. Wilkinson both edited the Proceedings and provided the new material.

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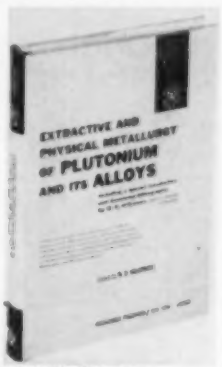

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editorial

An Empirical Art?

There exist prevalent beliefs that the only way to find ore-bodies is by prospecting in the field; the only way to discover a means of concentrating a particular ore is to test it by certain ore-dressing procedures; and the only way to extract a pure metal from a mineral concentrate is to test various schemes which have worked on other materials. A hundred years ago, these beliefs were correct for the vast majority of problems to be solved. Today, many of the problems in ore extraction are still solved by such empirical techniques, but the difficult and important problems yield only to the approach through basic science.

There should be no need to justify the approach to engineering problems through understanding the scientific bases of the phenomena involved. Nuclear power and the transistor are recent notable examples of the power of this method. Yet, in the field of ore extraction, tradition dies hard, and tradition has labelled the field an empirical art. While most people would be willing to admit that the prospector may be aided by certain basic principles of geology, and the extractive metallurgist by a general knowledge of chemistry and physics, very few people realize that this is no longer enough to solve the crucial problems of today.

There is a need for a sweeping revision of our concept of research on ore supply and extraction, and the new concept must be made understandable to all. The revised concept is simply stated: Important advances in geologic prospecting, ore dressing, and extractive metallurgy must be built on the results of fundamental research on, for example, the geochemistry of mineralization, the surface chemistry and physics of flotation, and the high-temperature inorganic chemistry of metals, metallic compounds, and their solutions.

Graduate study in ore dressing and extractive metallurgy fails to attract the number and quality of students and instructors needed to revitalize these disciplines. The science-oriented student chooses physical metallurgy in preference to extractive metallurgy because he sees only the empirical-art aspect of the latter field, and because financial support of project research is more available in physical metallurgy.

Science-oriented research in these fields is not an entirely novel concept that we must encourage to grow from the beginning. It has been well established in half a dozen graduate schools throughout the United States over the past 20 years, and also in some of the more advanced industrial research laboratories. The nucleus for growth of the concept is well established. It only remains to create the condition for more rapid growth.

HERBERT H. KELLOGG

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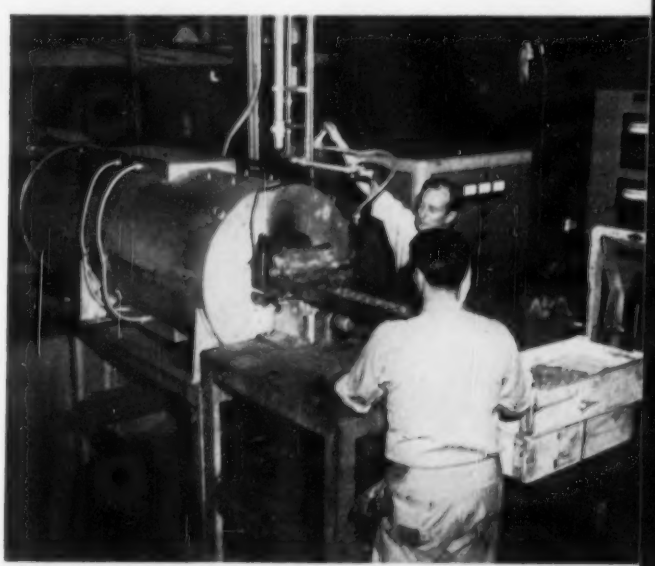

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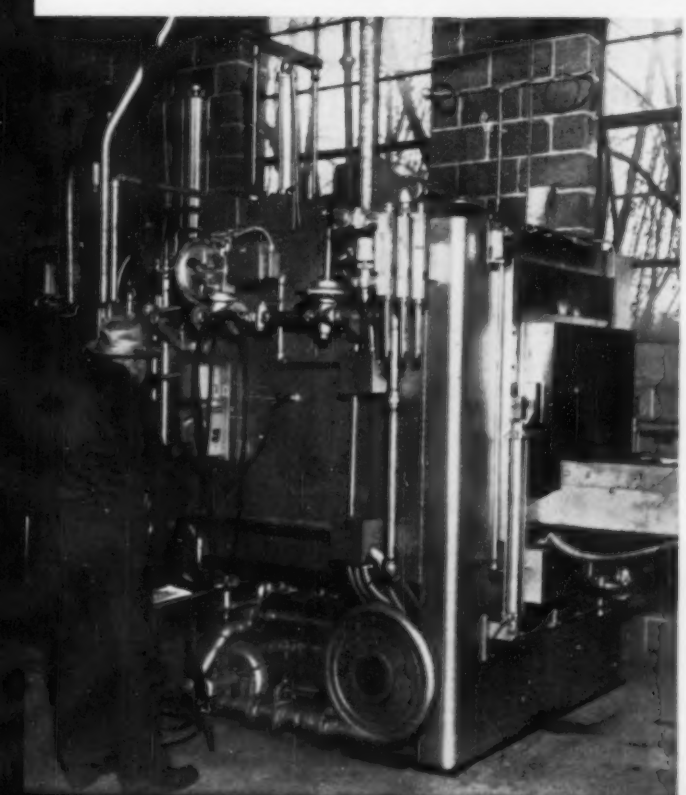

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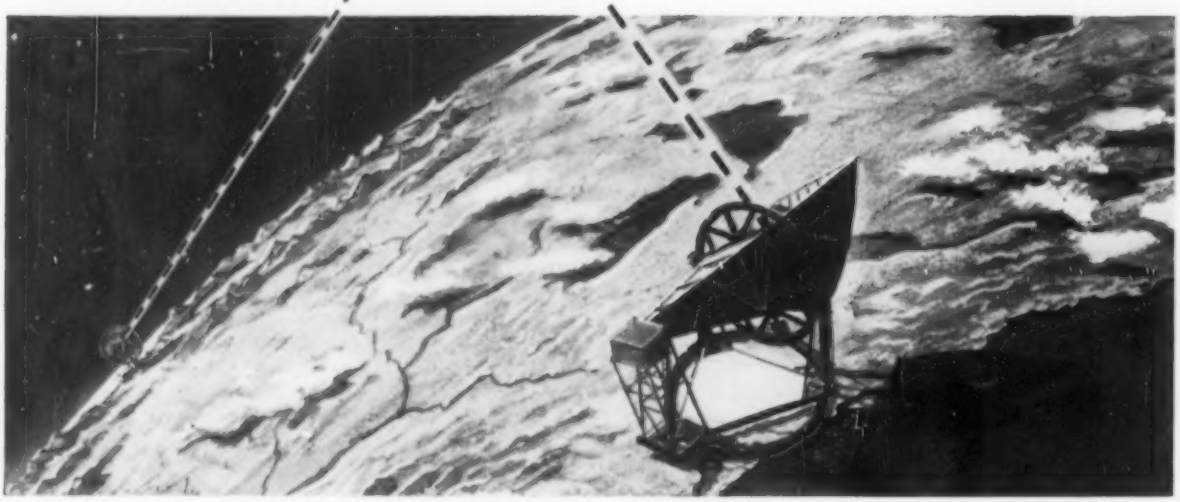

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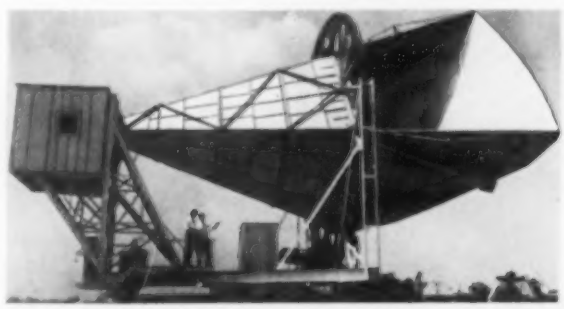
"Project Echo" foreshadows the day when numerous man-made satellites might be in orbit all around the earth, acting as 24-hour-a-day relay stations for TV programs and phone calls between all nations.

This experiment shows how Bell Laboratories, as part of the Bell System, is working to advance space communication. Just as we pioneered in world-wide telephone service by radio and cable, so we are pioneering now in using outer space to improve communications on earth. It's part of our job, and we are a long way toward the goal.



Giant ultra-sensitive horn-reflector antenna which received signals bounced off the satellite. It is located at Bell Telephone Laboratories, Holmdel, New Jersey.

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BERYLLIUM RESEARCH IN MISSILE DEVELOPMENT

Recent accelerated activity in beryllium research is reflected in the new uses for the metal in missile and space vehicles. The author presents a lucid account of the current achievements in the field of beryllium research.

by Clayton O. Matthews

After several years of applications development, another of the less common metals—beryllium—has progressed considerably in qualifying as an engineering material. Structural and physical property data are being obtained for the reliable design of selected flight structures for missiles, earth satellites, and spacecraft. Concurrently, studies of beryllium physical metallurgy and fabrication development have further increased confidence in its use and future potential. Utilization of beryllium metal in flight-vehicle structures has been pioneered by the Missiles and Space Division (LMSD) of the Lockheed Aircraft Corporation. When the present undertakings have been more completely realized, it is expected that the metal will be adopted more generally by the aerospace industry. The kind of information being obtained and some recent contributions of interest to metallurgists are reviewed in this discussion.

Although beryllium has been suggested occasionally for aircraft use during the past 25 yrs, it was not until the last decade that there was sufficient incentive to encourage the production of the metal and research on its properties. This technology, initially inspired by non-structural nuclear reactor interests, was complemented in the last several years by more demanding engineering requirements in nuclear fuel elements and aerospace vehicles. When this growth pattern is considered, it is apparent that initial product specifications—namely, for moderator-grade beryllium free from high cross-section neutron-absorbing elements—have little bearing on what is desirable for obtaining the optimum physical properties required in structures.

New space-age structural interests inspired metallurgists in a renewed search for sufficient scientific and technical understanding to provide improved formability and toughness in missile-quality beryllium. Unfortunately, experience has shown that further improvement of the brittle character of this metal may be difficult to attain even with the most imaginative and diligent scientific research. Numerous promising approaches await systematic and concerted treatment.

Low ductility is the prime deterrent to extensive use of beryllium in missiles and spacecraft. However, designers have compromised on this point in many instances in order to exploit the outstanding advantages of beryllium's exceptional modulus of elasticity, low density, elevated-temperature strength, and thermal properties. These properties can be appreciated by visualizing a metal having: the weight of magnesium, the stiffness of molybdenum, an elevated-temperature strength (weight basis) exceeding titanium, the oxidation resistance of aluminum, and total heat absorption to melting 6.5 times that of copper. Such a metal is uniquely suited to the environments of: flight in air or space, thermal effects in re-entry, efficient structural loading, and the radiation environments of nuclear reactors and outer space. Material properties such as these are very attractive to the missile designer and beryllium seems to fulfill his needs except for its fracture behavior. This particular aspect of beryllium especially deserves concentrated research effort.

With stringent application of structural mechanics, aeronautical engineers are finding that important missile components can be made from a low-ductility material, particularly if it exhibits considerable ductility at elevated temperatures. It is now known that the brittleness at ordinary temperatures can be alleviated by control of grain size and texture, composition, and heat-treatment condition in accord with traditional metallurgy. Thus, a degree of metallurgical confidence has arisen, which is reassuring since new metals are mysterious and suspect in a way which retards their full utilization.

Surprisingly little metallurgical effort has been expended on developing beryllium with improved fracture and impact properties at ordinary temperatures in comparison with that expended on any one of the specialized aircraft alloys based on aluminum, magnesium, titanium or steel.

The remarkable potential of beryllium for flight applications awaited an appreciation of its strategic importance in the exploration of space. Frequently quoted causes for delayed development have been scarcity, cost and toxicity, all of which are considered irrelevant reasons for delaying research and development of a strategic material. More widespread metallurgical experimentation with beryllium has been deterred particularly by: 1) delayed appreciation of the metal's thermal-structural potentialities; 2) limited availability of laboratories as a result of inadequate facilities for safe handling; 3) major research support from sources that permit only applications research and evaluation; and 4) hesitation by metallurgists who have considered

CLAYTON O MATTHEWS is a staff scientist in the LMSD Scientific Research Laboratory of the Lockheed Aircraft Corporation, Palo Alto, Calif. This paper was originally presented at the AIME Southwest Metals and Minerals Conference in Los Angeles in April, 1960.

earlier indications of the "inherent brittleness" of beryllium as final, without realizing that only modest improvements in fabricability and room-temperature ductility are required and appear feasible.

Flight structures

G. A. Hoffman[3] evaluated beryllium for aircraft and concluded that structural panels designed either by buckling or tension criteria weigh less than half as much as those made of conventional materials at both normal and elevated temperatures. This is based on the thought that a suitable product was available for such panels. In an economic analysis Hoffman determined that if beryllium could be used in transport aircraft, it would yield a significant savings per ton-mile of cargo—possibly to a cost of about $600 per lb of installed metal, well above manufacturing expectations.

In a comparison of structural advantages of materials, strength/weight factors alone are inadequate; the modulus of elasticity must also be considered. A realistic comparison can be made correctly based on the optimum design of a thin-walled structure designed by buckling criteria, such as a sheet-stiffener panel, where a typical loading is resolved as compression, shear and bending. This technique was used by Hoffman and by Riedinger and Foster[3] for comparing conventional materials up to 650°C as shown in Fig. 1. The weight was determined after optimizing a typical multi-web fin configuration for typical loading of Q = 2,220 lb per in. for the materials at given temperatures. The data illustrate the weight reduction realized by using beryllium.

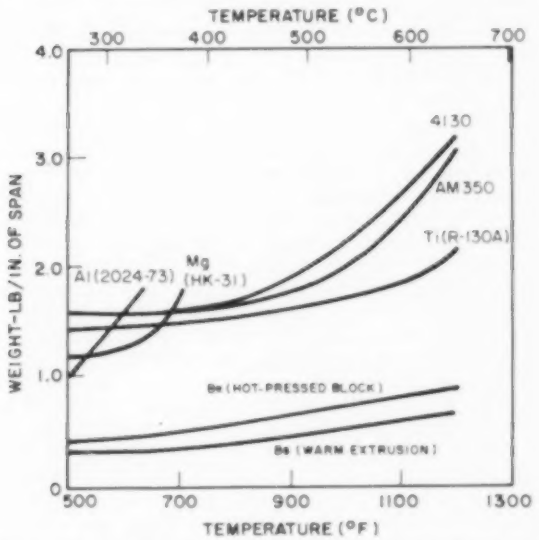

Fig. 1—Comparative relative weight of structural materials based on optimum design.

Applying this weight advantage to a 2-stage ballistic re-entry application[3] shows that each pound saved in the final payload section reduces the total weight of the vehicle by 42 lb. Similarly, in a 3-stage, liquid-propellant lunar-rocket application with a 1000-lb payload, a beryllium structure for the third stage would weigh about one-third as much as a steel structure and the total take-off weight is reduced by 210,000 lb, or 21 pct. Either the technical and economic advantage could be taken by this reduction, or the payload weight increased by 26.7 pct.

Based on a criteria of strategic importance and feasibility, it appears that emphasis on the use of beryllium structural components should be considered progressively in the following areas: 1) relatively simple shapes for missile payload sections, satellites, re-entry capsules, and spacecraft; 2) body structures, brackets, actuators, and equipment components in missile upper stages; and 3) transport aircraft structures. Until the brittleness problem has been resolved and test programs completed on design data, it is not certain to what extent beryllium will qualify as an acceptable structural material for the more complex configurations. The industry is presently engaged in the early stages of such a program from the standpoints of both structural evaluation and material development. Considerable success has been had in the first area.

LMSD projects

Earlier in the Lockheed program, several exploratory projects were jointly conducted with subcontractors on material development. Electron beam melting yielded ingots of good quality with reduction to sheet yielding a recrystallized product with properties presently at the low range for comparable sheet originating from powder. The preparation of pellets from quenched molten metal for subsequent consolidation appeared feasible. Direct rolling of powder produced a bendable sheet due largely to about 5 pct porosity which exhibited low tensile strength. Hot isostatic pressing of small cylinders has been demonstrated and typical properties obtained. Only the development projects directly applicable to current engineering component requirements are being conducted further.

Metallurgical research at Lockheed has been concerned with program direction, data searching[4], laboratory evaluation of products, property determinations, and general metallurgical investigations. Structures and test personnel have conducted extensive tests necessary for design purposes. The metallurgical and property studies will be highlighted in the remainder of this review. Such other important beryllium activities as fabrication and component development, environmental testing, and flight testing cannot be discussed at this time.

Electron bombardment melting

A technique for producing ingots 3 in. in diameter and up to 20 in. in length under high vacuum conditions by electron bombardment was successfully developed[5] and more than twenty ingots have been made in various experiments. A section of a typical ingot is shown in Fig. 2 in which the dendrites, curving from edge to center of the ingot, are sectioned across the smaller ingot dimension. Superior grain structure and machinability for cast beryllium was obtained along with freedom from ingot defects. The tensile strength, however, remained typically low for cast metal at about 20,000 psi with 0.1 to 0.2 pct elongation. Pechiney electrolytic beryllium is appreciably softer (R_B 65) than conventional induction-melted pebble (R_B 80) after electron bombardment processing (Table I). Purification occurs with more volatile elements Mg, Al, Si, while the more serious and abundant contaminants the interstitial elements C, O, N, H and the iron-group elements Fe, Ni, Mn, Cr are relatively unaffected. The oxygen content decreases, however

Table I Typical Composition and Hardness After Beryllium Electron Bombardment Processing

Melting Stock	O γ-Activation (ppm)	C γ-Activation (ppm)	H (ppm)	N (ppm)	Total Metallic Impurities (ppm)	Hardness Avg. R_B
Pechiney flake	38 ±8	164 ±49	195	71	400	65
Induction-melted ingot	51 ±10	540 ±108	56	54	3,200	80
Arc-cast ingot	—	—	77	170	5,500	82
Electron bombardment distillate	149 ±30	150 ±	47	60	1,800	72

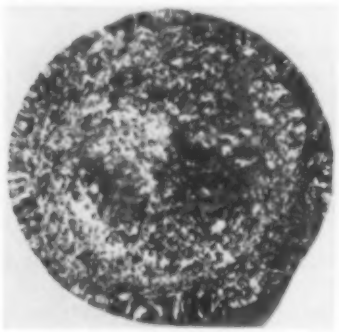

Fig. 2—Cross section of electron-bombardment melted, drip-cast beryllium ingot 3 in. in diam.

by segregation at the ingot surface. In addition to obtaining improved cast ingots, a method has been demonstrated for consolidating beryllium from a future super purity source without contamination.

In electron-bombardment melting experiments involving composition, it was found that reduction of oxygen by carbon addition was ineffective. Pechiney flake containing 5000 ppm total impurities —assaying principally as oxygen—was purified to 1780 ppm impurities by electron-bombardment consolidation. Distillation by electron-bombardment heating, collection of the distillate on a hot metal surface, and consolidation in the same furnace reduced the total metallic impurities markedly from 7270 ppm to 1800 ppm, with a decrease in hardness of R_B 72. Purification by distillation appeared to be attractive as a potential commercial process.

Typical electron-bombardment cast ingots have been extruded and rolled at the Beryllium Corporation with encouraging success. Grain refinement has resulted in recrystallization and 90-mil sheet exhibited up to 39,000 psi tensile yield strength with 2.5 percent elongation. Further improvement in properties and production economy is anticipated.

Radiation activation analysis

It soon became apparent that a new method would be essential for the evaluation of oxygen at less than 200 ppm. In cooperation with nuclear physicists, the γ-activation analysis technique was developed which has demonstrated capability in the important trace impurity range for O, C, and N[6, 7, 8]. The specimen is activated by gamma rays using the 40-mev linear accelerator at Stanford University. As the resultant impurity element activities decay by positron emission, they are quantitatively detected by coincidence counting. The method has been used to detect 10 to 5,000 ppm O and 100 to 25,000 ppm C with a precision of 20 pct. In the absence of interfering activities as in very pure beryllium, the minimum amount of oxygen and carbon detectable is expected to be 1 ppm with a precision of 5 pct. Nitrogen content can be estimated at trace concentrations when the iron and copper contents of the sample have been determined spectrographically, since their activities combine with N when counting their decay emission. The technique is used for measurements in the development of high purity beryllium and as a standard reference method for evaluating other analytical techniques for oxygen. It is also the most reliable method for carbon in beryllium in the 10 to 1000 ppm range.

Alloying and casting

Extensive investigations have been made of the effect of alloying on beryllium cast structures[9]. These investigations have been undertaken with the commercially available, magnesium-reduced beryllium of 98 to 99 pct purity, or with beryllium flake of approximately 99.6 pct purity. Considering the extremely small solubility limits of most metallic and interstitial alloying elements with beryllium, it is apparent that the solubility limits for many of the elements have been approached or exceeded by the amounts present as impurities. Because of this the effects of intentionally added elements are masked by the effects of residual elements. It appears that until high-purity beryllium of less than 100 ppm total impurities becomes available for experimental purposes, the most favorable area of investigation for producing cast beryllium of improved properties is probably that of controlling the solidification process to produce a fine-grained, equiaxed structure.

The effects of Si, Ag, and Al on Be alloyed with 1 pct Ti on the percentage of equiaxed grains are shown in Fig. 3. Of the several hundred beryllium-rich alloy buttons made and evaluated by preliminary screening tests, those showing potential for improved mechanical and fabricating properties are binary and ternary combinations containing 2-4 pct Si, 0.1-1 pct Ag, 0.1-1 pct Ti, and 2-4 pct Al. In addition to grain structure other criteria in the screening evaluation have been hardness and susceptibility to microfracture. Small ingots are being vacuum cast for more thorough evaluation of properties, casting characteristics, and fabricability studies. Excellent castability in a 1 in. by ½ in. by 6 in. chill mold for example, was observed with an alloy containing 0.5 Ti and 0.1 Ag by weight as compared to an identical casting of unalloyed beryllium. (Fig. 4) If an equivalent structure can be retained in larger ingots a more economical step in the process for wrought mill products, perhaps with better ductility or fracture behavior, may be found.

The prospect of enhancing ductility by solid solution alloying or possible combination with interfering impurities has long been discouraging. The present approach of controlling the grain size and orientation texture during casting and subsequent deformation is definitely promising. Also, permanent mold and centrifugal casting appear feasible for the first time as satisfactory and economical methods for producing low impact strength and ductility components.

Corrosion in air

Beryllium is known to be resistant to air to about 850°C but at higher temperatures especially to the melting point 1285°C, the reaction mechanism and kinetics have been uncertain. Accordingly, this reaction and to some extent the reaction in oxygen and nitrogen separately have been studied[10].

An induction period has been observed in air which is 10 min at 1115°C and 2 min at 1255°C. The experimental results indicate that the corrosion of beryllium in air follows a linear rate, and an Arrhenius plot of the reaction rates (Fig. 5) shows the reaction to be temperature dependent with an activation energy of 1.1 x 10^5 cal per mole in the range 930° to 1065°C and 1.06 x 10^4 cal per mole in the range 1056° to 1295°C. The determination has been made both gravimetrically and by pressure change in a recording monometric system. Further precision will soon be obtained using an automatic recording balance.

Electrical resistivity

Electrical resistivity of beryllium is being studied as it pertains to the effects of impurities and the relationship with thermal conductivity[11]. The data reveal little correlation with nonmetallic or total impurity content, verifying that the solubility limits of the nonmetallics have been exceeded and are present as second phase particles such as beryllium oxide. The resistivity varies clearly as a function of metallic impurity content. For example, in hot pressed block material the resistivity increased at 4°K (−269°C) from 0.5 microhm-cm at 0.1 weight pct metallic impurity to 5.5 microhm-cm at 0.8 pct metallic impurity. Similarly at 300°K (27°C) the resistivity increased respectively from 4.5 microhm-cm to 10 microhm-cm.

An attempt to develop a universal expression relating electrical and thermal conductivity of various grades of beryllium over its solid state temperature range has not been possible thus far, but may be determined above 700°C. Between 100° and 700°C the resistivity of specimens from different lots vary by an amount as great as 20 microhm-cm (Fig. 6). This variation may depend on the degree of precipitation of impurities according to prior thermal histories, which should become clarified when studies of the latter have been completed.

Some variation in thermal conductivity also occurs in this 100° to 700°C range. It is also in this range that beryllium experiences a rapid increase in ductility with temperature to a maximum at about 450°C falling off to a minimum at 750°C. This may similarly be related to aging and precipitation effects. When studies of these microstructural changes have been completed, beryllium's mechanical and physical property behavior may be much more readily correlated by mathematical relationships.

Mechanical properties

Extensive mechanical property and structural test data are being obtained on hot-pressed block, cross-rolled sheet, and hot upset beryllium and subsequently collated for a beryllium design data book. The test program is yielding such mechanical property information as strengths in tension and compression, short-time creep, strength in shear and bearing, torsion strength, modulus of rupture, elastic modulus in compression, fatigue, surface damage effects, buckling data, and bi-axial loading properties.

A reliability analysis reporting the percentage probability of predicted tensile properties for hot-pressed block in the pressing direction is summarized in Fig. 7. Properties transverse to the pressing direction show greater spread and the tensile strengths are generally somewhat less. While the spread in the data indicates nonuniformity and anisotropy in this type of beryllium product, the variation may be partially attributable to testing technique, surface damage, composition, thermal history, and other factors to which the metal may be sensitive. While some of these factors are carefully controlled, further technical progress should narrow this reliability margin.

In other tests, no significant creep was observed below 400°C in less than 3 min. A stress of 25 pct of the ultimate can be maintained for almost 15 min at 675°C but for less than a minute at 815°C. Shear

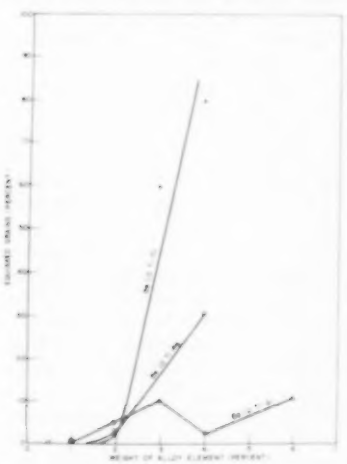

Fig. 3—Effect of Si, Ag, and Al on pct of equiaxed grains in beryllium alloyed with 1 pct Ti. (From Ref. 9).

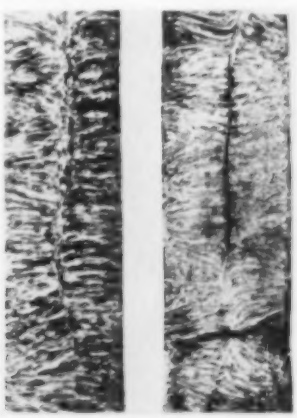

Fig. 4—Beryllium alloy casting containing 0.1 Ag and 0.5 Ti (left) chill cast in a 0.5 in. by 1 in. mold showing freedom from defects obtained with eutectic-type solidification; typical centerline shrinkage void was obtained in similar ingot without alloying (right).

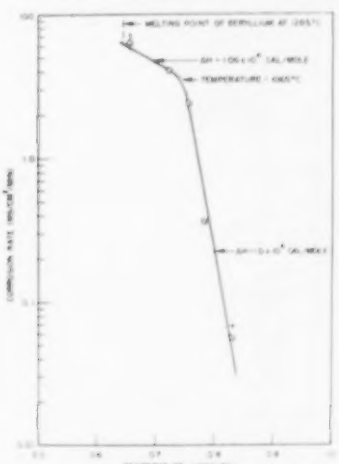

Fig. 5—Arrhenius plot of corrosion of beryllium in air at near atmospheric conditions. (From Ref. 11).

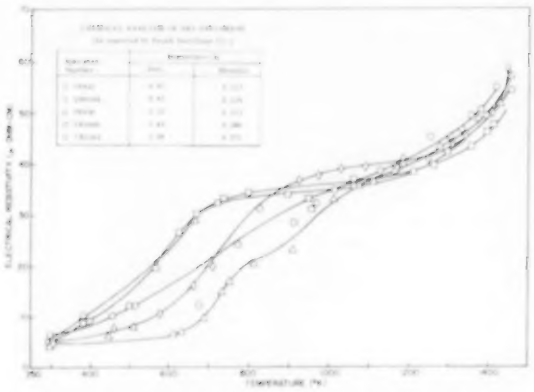

Fig. 6—Variation in electrical resistivity in different lots of beryllium as a function of temperature (From Ref. 11).

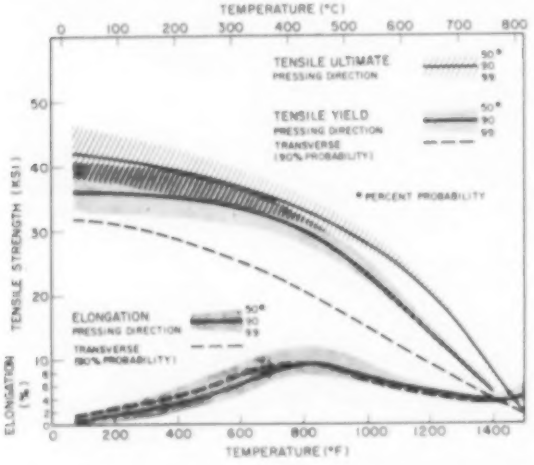

Fig. 7—Tensile strength and elongation of hot-pressed block beryllium. The lines represent 90 pct probability that a given sample will have an equal or better value than indicated. Shaded areas show probability range from 50 to 99 pct. Strain rate equals 0.005 in./in./min.

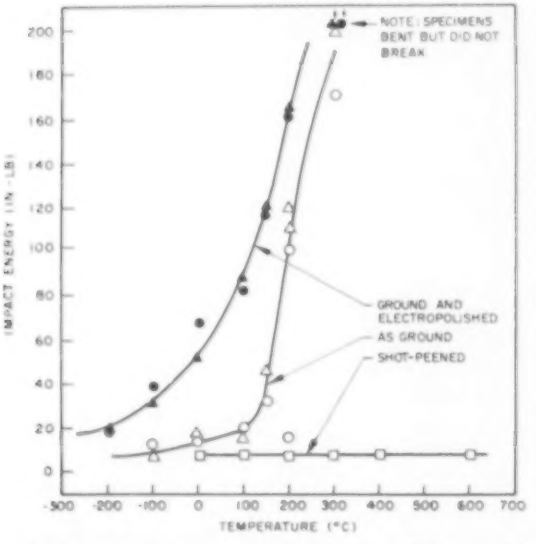
NOTE: SPECIMENS BENT BUT DID NOT BREAK
GROUND AND ELECTROPOLISHED
AS GROUND
SHOT-PEENED
IMPACT ENERGY (IN.-LB)
0 20 40 60 80 100 120 140 160 180 200
-500 -200 -100 0 100 200 300 400 500 600 700
TEMPERATURE (°C)

Fig. 8—Impact resistance vs. test temperature for hot-upset plate specimens 0.25 in. thick by 0.5 in. wide.

strength is about equal to tensile strength at room temperature and to about 80 pct of tensile strength at 540°C. Modulus of rupture ranged from 50,000 to 70,000 psi depending on the geometry of the beam cross section with little change to 200°C and decreasing to zero at 760°C. Both room- and elevated-temperature tensile properties are significantly increased by increasing oxide content up to several percent beryllium oxide. The fatigue properties were found to be unique in that the fatigue endurance limit is the same as the static strength, and the S-N curves are horizontal lines with all surface conditions[12]. No susceptibility to stress corrosion cracking was observed[13]. The design data will be generally available when completed.

Surface damage in rolled sheet surface finished by machining operations was evaluated by tests of tension, notch tensile, fatigue and impact strengths[12]. Strengths were generally reduced by surface grinding, milling, and particularly shot peening. Only after chemical removal of several mils from each surface were optimum values obtained. Metallographic examination of sheet cross sections revealed frequent subsurface fracture with all machining methods—the principal rupture plane being about 2 mils beneath the finished surface. Extensive twinning also occurs in the first several mils near the surface even with light grinding or milling cuts. This may also contribute to a mechanism for fracture initiation.

The most discriminating test for evaluating the effect of surface damage is by impact testing over a range of temperatures and the determination of the ductile-brittle transition temperature (Fig. 8). The impact strength with unnotched Charpy-type specimens—very low in the unetched condition (less than 1 ft-lb)—is increased by removing damaged surface material by an average of 5 ft-lb to the transition temperature. Since surface damage is prevalent in all untreated beryllium materials, special surface preparation of all structural components is recommended.

Residual stress near the surface of rolled sheet was measured by the distortion of a panel as thin layers of one surface were removed chemically. Micrometer measurements of the panel deflection were computed to residual stress by the Treuting-Read method. It was found that residual compressive stress is present at the surface of cross-rolled sheet measuring 24,000 psi as rolled, 39,500 psi as milled, and 16,500 psi as ground. Beneath the surface, the stresses decreased linearly to 10,000 psi at a depth of 10 mils. Clearly, the presence of forces of this magnitude near the surface of the material is highly significant when combined with applied stresses in structures.

Attempts to measure the force required to propagate a crack in beryllium and the fracture velocity by conventional methods (using centrally slotted panels) have been prevented by the inherent difficulty in introducing incipient cracks at the tips of the slots. Fracturing of the full width of the panel occurred while fatigue loading to initiate the crack, and indentation loading resulted in pre-cracking of a minimum of one-third of the panel. Observations that the effective remainder of the panel sustained about 45 pct of the potential load carrying capacity and that the fracture rate was instantaneous can be taken as indicative of the importance of beryllium's crack propagation behavior in its use in structural applications.

Joining

Mechanical joints and silver brazing are being used satisfactorily to assemble components, and it appears that earlier difficulties with fusion welding are being mitigated. Although coarse grain structure and cracking have generally yielded poor weld properties, better results are now being obtained. Recent arc welding development using automatic filler wire feed at the Brush Beryllium Company is showing promise. In addition, it appears likely that the utilization of the improved solidification and structure of the new LMSD alloys, shown in Fig. 4, may contribute a major step in beryllium welding.

A feasibility study of electron beam welding indicated that although the technique remains to be demonstrated for structural applications, welding at 25 to 30 kv with a sharply focused beam will be practical and capable of producing reliable welds in commercial beryllium. Sheet and plate up to ½ in. in thickness with unusually narrow fusion zones —less than ⅛ in.—are possible. This provides the rapidly quenched fine-grain structure in a minimal fusion zone especially necessary for beryllium. Vacuum systems integrated with the requisite electron-bombardment units have been developed by several manufacturers.

Summary

The limiting problems with beryllium are primarily related to ductility with ramifications in low impact resistance, catastrophic crack propagation, limited formability, weld brittleness, and sensitivity to surface damage. Means have been found to alleviate these shortcomings and the considerable elongation at elevated temperatures has been a saving feature for missile application.

Many variations in beryllium cast, sintered, rolled, and extruded products have been made with properties covering a wide range easily encompassing the properties required for design purposes. Yield strengths ranging from 40 to 70 ksi are adequate; room temperature elongation of 5 to 10 pct in warm-rolled sheet tensile specimens has been obtained; crack propagation in hot upset sheet is retarded by less-preferential distribution of fracture planes; and powder rolled sheet with several percent porosity can be formed to a sharp radius. No single beryllium product combines the best properties in one material, but their occurrence independently suggests that better combinations may be possible.

If attempts at purification and orientation control in wrought beryllium grain structures are ultimately unsuccessful, ductility improvement by such alternate means as developing a dispersion structure shows promise. Such a microstructure may consist of optimized microporosity, possibly infiltrated with a ductile metal. On the other hand it may be obtained by chill quenching molten beryllium containing an alloy solute to obtain ultrafine particulates dispersed in beryllium, followed by consolidation of the quenched pellets by extrusion and rolling. It should be noted that present commercial powder metallurgy beryllium is dispersion strengthened with more than one percent beryllium oxide. There are undoubtedly other similar techniques also awaiting proper trial.

The entire area of thermal treatment of commercially pure metal or modified compositions selected for this purpose has barely been touched. Work at the Brush Beryllium Company[16] has shown that a 750°C heat treatment for 20 min. with air or furnace cooling doubled the elongation at 650°C in the high temperature ductility valley. Mash[18] observed that solution treating at 800°C, oil quenching and aging at 300°C increased the yield strength from 32 to 44 ksi. The investigation of thermal treatments in conjunction with composition studies is overdue.

Further optimism for structural beryllium is justified, particularly for missile and space use, but no implication is intended that beryllium lends itself easily to metallurgical treatment. It must be appraised for what it is, not a high-volume mill product for all light metal applications, but as another specialty product suited to unique nuclear and aerospace-flight components. Design engineers agree that the present need in products is greater uniformity permitting predictable and reliable mechanical behavior. Although upgrading the room-temperature ductility to about 5 pct elongation is greatly desired by designers, it is pointed out that an increase of only 1 pct elongation would improve performance at this temperature more than 50 pct in the most commonly used product.

Improving ductility of beryllium is not an easy task but some improvement appears technically possible and the incentives are sufficiently rewarding to recommend a more intensive effort. Fabrication of new beryllium products should be undertaken and an intensified research effort made to ascertain whether the metal's brittleness is due to its inherent physical nature. The following approaches should be encouraged: 1) research of basic factors responsible for ductility in beryllium; 2) development of technology for processing wrought and cast products and fabrication techniques permitting the greatest utilization; 3) achievement of product and property uniformity through process control that satisfies realistic engineering specifications; and 4) continued application in increasingly complex flight structures.

Given an adequate opportunity, a new metal technology can mature and contribute to our national satellite and space travel progress and prestige more significantly than is commonly anticipated.

References

[1] Materials Advisory Board, Report of the Physical Metallurgy Group of the Panel on Beryllium, Report No. MAB-129-M, National Academy of Sciences, National Research Council, 11 March 1958.

[2] G. A. Hoffman, Could Beryllium be Used as a Structural Material?, *Materials and Methods*, Feb. 1957.

[3] L. A. Riedinger, and R. E. Foster, Beryllium Applications in Structural Design, *Astronautical Sciences Review*, July-Sept. 1959.

[4] W. G. Bradshaw, Beryllium—A Search of the Unclassified Literature, LMSD-2260, Lockheed Aircraft Corporation, Sunnyvale, Calif. Nov. 1957.

[5] H. T. Sumsion and C. O. Matthews, High Vacuum Melting of Beryllium by Electron Bombardment, *Vacuum Metallurgy*, New York University Press, 1959.

[6] Gamma Activation Measures Oxygen, Carbon in Beryllium, *Nucleonics*, Vol. 17, No. 7, 1959.

[7] W. G. Bradshaw, R. Johnson and D. B. Beard, "Beryllium Analyzed for Trace Impurities by Gamma Ray Activation, ACS paper, Sept. 1959.

[8] W. G. Bradshaw, R. Johnson and D. B. Beard, Beryllium Analyzed for Trace Impurities by Gamma Ray Activation, LMSD-288231, Lockheed Aircraft Corporation, Sunnyvale, Calif., Jan. 1960.

[9] D. Crooks and H. T. Sumsion, Grain Refinement in Be by Alloying, LMSD-288233, Lockheed Aircraft Corporation, Sunnyvale, Calif., 1960.

[10] W. G. Bradshaw and E. S. Wright, High-Temperature Corrosion of Beryllium in Air, LMSD-288232, Lockheed Aircraft Corporation, Sunnyvale, Calif., Jan. 1960.

[11] E. S. Wright and J. Ho, Electrical Resistivity of Beryllium, LMSD-288218, Lockheed Aircraft Corporation, Sunnyvale, Calif., Feb. 1960.

[12] C. O. Matthews, M. I. Jacobson, W. Jahsman, and W. V. Ward, Crack Propagation and Surface Damage in Beryllium Sheet, Final Report WADC Contract AF-33(616)-5978, Jan. 1960.

[13] C. M. Parker, Stress Corrosion Cracking of Beryllium, LMSD-49735, Lockheed Aircraft Corporation, Sunnyvale, Calif., May 1959.

[14] R. G. O'Rourke, J. N. Hurd, K. G. Wikle, W. W. Beaver, Mechanical Properties of Reactor-Grade Beryllium at Elevated Temperatures, Report No. COO-312, Brush Beryllium Company, Aug. 1956.

[15] D. R. Mash, Ageing Effects in Commercially Pure Beryllium, *AIME Transactions*, Vol. 203, No. 11, Nov. 1955.

FUSED SALT ELECTROREFINING OF BERYLLIUM

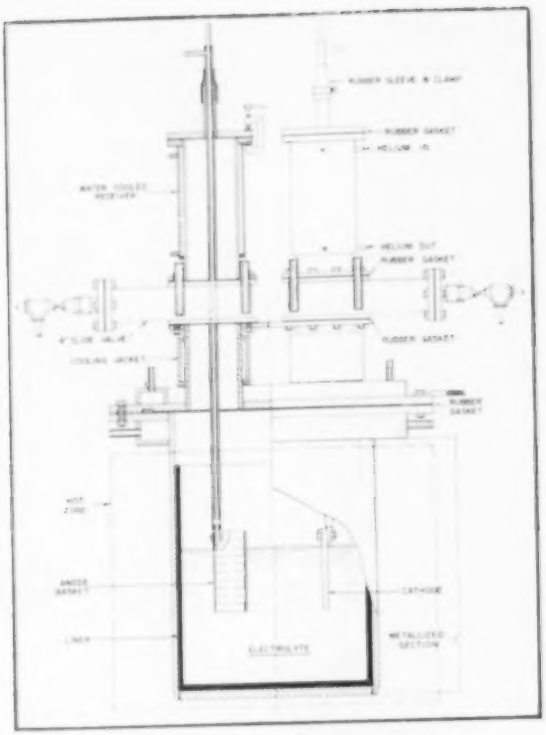

Fig. 1—The fused-salt electrorefining cell.

The US Bureau of Mines has achieved laudable progress in its striving to attain high-purity beryllium. The following is a discussion of the Bureau's work on the fused salt electrorefining of beryllium.

by M. M. Wong,

R. E. Campbell, and D. H. Baker, Jr.

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BERYLLIUM has long been recognized as a metal with important physical properties for use in space vehicles and in the nuclear field. The purpose of our work is to prepare beryllium in high-purity form useful for physical metallurgy studies and to determine the electrorefining characteristics of low grade metal—to furnish information in applying this technique to reclaiming scrap metals.

The success with fused salt electrorefining of titanium at the Federal Bureau of Mines Metallurgy Research Laboratory, Boulder City, Nevada, led to the application of this electrorefining technique to beryllium and other metals. This report covers the early results of studies on electrorefining beryllium which are still underway.

The apparatus, shown in Fig. 1, was designed to permit the electrorefining operations to be performed under an inert gas, thus preventing atmospheric contamination. The cell consisted of an electrolyte chamber and a cover assembly.

The electrolyte chamber was constructed of mild steel, 12 in. in diam and 30 in. long—the inside lined with a graphite crucible and the outside metallized with a protective coating. The chamber was heated in an electric resistance furnace.

The cover assembly contained two receiving chambers to accommodate the anode and the cathode. Fittings for helium, manometer, bubbler, and vacuum pump were provided in these chambers. A water-cooled slide valve was installed between each receiving chamber and the electrolyte chamber.

The electrode leads were made of copper tubes, internally water-cooled, and were inserted into the cell through fittings provided in the covers of the receiving chambers. An iron rod was used as

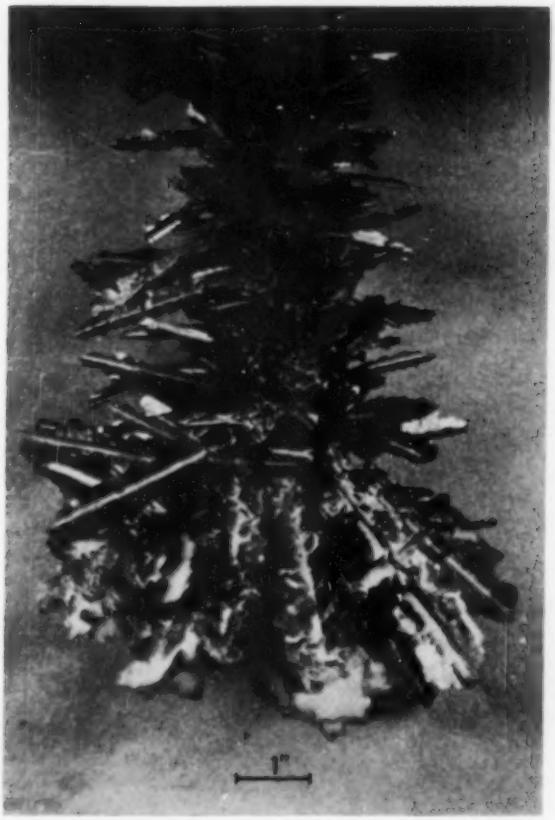


Fig. 2—Cathode deposit as removed from cell.

Fig. 3—Beryllium dendrites.

the depositing cathode and an iron basket containing crude beryllium metal as the anode. Direct current for electrolysis was furnished through a selenium rectifier.

Procedure

The cell was assembled and tested for leaks under both vacuum and pressure. When it was proven to be leak-free, a total of 50 lb of cp grade LiCL and KCL of eutectic composition was added. After the cell was purged under vacuum at 200°C and 400°C for 24 hrs, the cell, backfilled with helium, was then heated to the operating temperature—450°C or 550°C.

Atmospheric contamination in the cell must be kept to a minimum. To achieve this the slide valves between the electrolyte chamber and the receiving chambers were closed when the receiving chambers were opened to the outside for the removal or introduction of material during operation. The receiving chambers had to be sealed and purged of air before the slide valves to the electrolytic chambers could be reopened.

The $BeCl_2$ component of the bath was produced *in situ* by reacting HCL with beryllium metal ($2HCL + Be \rightleftarrows BeCl_2 + H_2$) in a graphite tube partially immersed in the molten salt. $BeCl_2$ passed from the tube and was dissolved in the molten salt, while the H_2 was vented from the cell through an oil bubbler. When the amount of HCL, calculated to yield the desired concentration of $BeCl_2$ in the bath, was consumed, the chlorination apparatus was removed, and the electrodes were installed for electrolysis.

At the completion of a deposition cycle, the cathode deposit was allowed to drain and then cooled to room temperatures in the receiving chamber. After the metal was stripped from the cathode, it was broken up and leached in a dilute HNO_3 solution, filtered, washed, rinsed with acetone, and dried in a hood.

Results

Data of typical tests are shown in Table I. These tests may be divided into three groups: (1) 56.9 mole pct LiCL-39.6 mole pct KCL-3.5 mole pct $BeCl_2$ bath at a temperature of 550°C, (2) same bath at a temperature of 450°C and (3) 49.3 mole pct LiCl-34.2 mole pct KCl-16.5 mole pct $BeCl_2$ bath at a temperature of 550°C.

Table II shows the analyses of the anode feed and the refined metals. The analytical methods employed follow: for oxygen and hydrogen, vacuum fusion using the platinum bath technique; for chloride, the silver chloride gravimetric method; for nitrogen, the Kjeldahl method; for carbon, the combustion method; and for metallic elements, spectrochemical method. Oxygen, magnesium and beryllium in the anode feed were determined by the following methods respectively: acid solution and determination of the quantity of the residual beryllium oxide, precipitation of magnesium as magnesium phosphate and conversion to pyrophosphate, and precipitation of beryllium hydroxide and conversion to oxide. The adaptation and development of analytical procedures were carried on concurrent with the progress of the electrorefining work. Progressive refinements of the determination limits, particularly in the spectro-

chemical analyses, as shown in the results of the later runs over those of the early runs, are in evidence.

Deposit

The deposited beryllium metal, depicted in Fig. 2 and 3 were steel gray in color and in the form of thin, flat platelike crystals. Long fibers of platelets were produced in the bath containing 3.5 mole pct $BeCl_2$ and large lamellae were produced in the bath containing 16.5 mole pct $BeCl_2$. Increasing cell voltage (also cathode current density)—somewhat decreased the size of the crystals. Crystals produced at the bath temperature of 450°C were noticeably less adherent to the cathode than those produced at 550°C.

Stability of $BeCl_2$ in Bath

Sublimination of $BeCl_2$ from the bath containing 3.5 mole pct $BeCl_2$, both at 450°C and 550°C was negligible and bath samples taken throughout the series of tests showed that the $BeCl_2$ content remained very constant. On the other hand, sublimination of $BeCl_2$ from the bath containing 16.5 mole pct $BeCl_2$ at 550°C. was appreciable, and bath samples showed that the $BeCl_2$ content decreased as the tests progressed.

Electrorefining

The metal products showed that electrorefining was achieved as all impurity elements—except carbon—that were found in the anode feed were reduced. Magnesium, nickel, aluminum, and chromium were reduced as to be almost undetectable by spectrochemical analysis. Oxygen, silicon, iron and nitrogen were also appreciably reduced. Generally some degree of reduction was indicated for manganese and copper, but electrorefining appeared a poor solution for these impurity elements. Some increase in carbon was indicated; however, this result might have been affected by the use of a graphite crucible as the electrolyte container.

Notwithstanding the fact that the determination limits were varied as the tests progressed, analyses of the metal products appeared to indicate that generally better electrorefining was obtained from the bath with 16.5 mole pct $BeCl_2$ at 550°C, than the one with 3.5 mole pct $BeCl_2$ at the same temperature. The operating temperature of 450°C with the bath containing 3.5 mole pct $BeCl_2$ gave better results in reducing metallic impurities than the same bath operating at 550°C.

The purity of the metal produced in the later runs, when analytical procedures were improved, compares favorably with that of high-purity beryllium produced from other sources as reported in the literature. In general, our metal appears to be lower in oxygen, magnesium, aluminum, nickel, and iron; higher in chloride, manganese, and copper; and of the same level in silicon and nitrogen.

References

[1] J. A. Gurklis, J. G. Beach, and C. L. Faust: Electrodeposition of Beryllium, Thorium, and Zirconium from Fused Salt Baths. *AEC Report BMI-781*, Nov. 4, 1952, 20 pp.

[2] K. Illig: The Production and Uses of Beryllium. *Trans. Am. Electrochem. Soc.*, vol. 54, 1928, pp. 53.

[3] B. R. F. Kjellgren and C. B. Sawyer: Method of Producing Metallic Beryllium. U. S. Patent 2,188,904, Feb. 1940.

[4] C. S. Pearsall: Effect of Oxygen on the Ductility of Beryllium Prepared by High-Vacuum Distillation. *MIT-1104*, Dec. 4, 1952, pp. 6.

[5] Siemens Company, The: Beryllium, Its Production and Application. Translated by R. Rimach and A. J. Michel. *Chemical Catalog Company, Inc.*, N. Y., 1932, 331 pp.

[6] H. T. Sumsion and C. O. Mathews: Electron Bombardment Melting and Casting of Beryllium. *L.M.S.D.-48330*, Nov. 7, 1958, pp. 35.

[7] J. M. Tien: Preparation of Beryllium Powder. *Trans. Electrochem. Soc.*, 1946, vol. 89, pp. 237.

[8] C. E. Windecker: The Production of Beryllium by the Electrolysis of Beryllium Chloride. *The Metal Beryllium*, edited by D. W. White, Jr. and J. E. Burke, *Am. Soc. Metals*, Cleveland, Ohio, 1955, pp. 102.

[9] M. M. Wong, F. R. Cattoir, and D. H. Baker, Jr.: Electrorefining Beryllium: Preliminary Studies. *Bureau of Mines Rept. of Investigations 5581*, 1960, 9 pp.

Table I. Data of Typical Tests

Run No.	Bath, mol. pct	Temp. °C	Cell voltage	Average amperage	Cathode current density,[1] amp/sq ft	Metal, grams	Cathode current efficiency, pct
1	56.9 LiCl, 39.6 KCl, 3.5 $BeCl_2$	550	0.25	6.5	110	19.0	80
2	56.9 LiCl, 39.6 KCl, 3.5 $BeCl_2$	550	0.49	14	200	12.5	92
4	56.9 LiCl, 39.6 KCl, 3.5 $BeCl_2$	550	0.67	23	340	15.6	94
6	56.9 LiCl, 39.6 KCl, 3.5 $BeCl_2$	550	1.04	28	410	19.5	92
8	56.9 LiCl, 39.6 KCl, 3.5 $BeCl_2$	450	0.49	11	160	27.8	89
10	56.9 LiCl, 39.6 KCl, 3.5 $BeCl_2$	450	0.71	12	330	10.0	84
12	56.9 LiCl, 39.6 KCl, 3.5 $BeCl_2$	450	1.17	21	300	13.8	88
14	49.3 LiCl, 34.2 KCl, 16.5 $BeCl_2$	550	0.31	8	115	15.8	70
16	49.3 LiCl, 34.2 KCl, 16.5 $BeCl_2$	550	0.58	12.5	180	10.8	87
18	49.3 LiCl, 34.2 KCl, 16.5 $BeCl_2$	550	1.08	29	425	13.6	62

[1] Based on initial cathode area.

Table II. Analyses of Anode Feed and Electrorefined Metal
Analysis (in pct)

Material	Be	Cl	O	N	H	C	Al	Co	Cr	Cu	Fe	Mg	Mn	Ni	Si
Anode feed	94.07	—	3.27	0.007	—	0.011	0.1-0.5	0.003	0.011	0.01-0.1	0.32	2.66	0.015	0.05	0.1-1.0
Electrorefined Metal:															
Run no. 1	—	0.23	0.045	0.001	0.001	0.029	0.004-0.03	<0.005	<0.006	0.01-0.1	0.08	<0.02	0.001-0.01	<0.002	<0.006
4	—	0.26	0.043	0.002	0.002	0.047	0.004-0.03	<0.005	<0.006	0.01-0.1	0.03	<0.02	0.032	<0.002	<0.006
6	—	0.26	0.038	0.004	0.001	0.030	0.004-0.03	<0.005	<0.006	0.001-0.01	0.035	<0.02	0.06	<0.002	0.008
8	—	0.45	0.037	0.007	0.006	0.030	0.004-0.03	<0.005	<0.006	0.001-0.01	0.029	<0.02	0.045	<0.002	0.006
10	—	0.42	0.045	0.01	0.002	0.020	0.004-0.03	<0.005	<0.006	0.001-0.01	0.035	<0.02	0.001-0.01	<0.002	<0.006
12	—	0.29	0.061	0.003	0.004	0.020	0.004-0.03	<0.005	<0.006	0.001-0.01	0.028	<0.02	0.001-0.01	<0.002	0.009
14	—	0.19	—	—	—	—	<0.001	<0.002	<0.006	0.013	<0.002	<0.0003	0.0048	<0.002	0.0021
16	—	0.11	—	—	—	—	<0.001	<0.002	<0.006	0.010	<0.002	<0.0003	0.008	<0.002	0.02
18	—	0.10	—	—	—	—	<0.001	<0.002	<0.006	0.009	<0.002	<0.0003	0.007	<0.002	0.0021

—, not determined.
<, below determination limit and, in most cases, also below detection limit.

Other metallic elements that were not detected, or only occasionally detected, include: Ag, B, Bi, Ca, Mo, Pb, Sb, Sn, Sr, Ti (except in Runs 7, 9, 10 and 12 where samples showed 0.015 to 0.02 percent of this element, probably caused by laboratory contamination), V, Zn and Zr.

RECENT ADVANCES IN BERYLLIUM METALLURGY

Sound metallurgical approaches toward understanding, as well as eliminating, the persistent problem of brittleness in beryllium metal are described in this paper.

by S. H. Gelles, J. J. Pickett, and A. Wolff

BERYLLIUM is a metal having very attractive properties for nuclear and space applications. These properties include a high modulus of elasticity, low neutron absorption cross section, and low density. The major deterrent to its more extensive use is its limited ductility. Investigators at Nuclear Metals Inc. have focused their attention on the brittleness problem for the past few years and have attacked the problem along three lines: 1) an effort to produce a crystal structure that is more ductile than the crystal structure (hexagonal close-packed) normally met in beryllium at room temperature, 2) a program aimed at discovering the cause of beryllium brittleness with special attention to the effect of impurities, and 3) efforts to produce a more ductile material by control of the preferred orientation in material fabricated from powder beryllium. In this paper the first two approaches are reviewed to show what progress has been made up to the present time, and what goals are contemplated for the future.

Retention of the High Temperature bcc Phase to Room Temperature

The first program to be described* is aimed at retaining the high-temperature phase present in pure beryllium to lower temperatures, hopefully to room temperature.

Work by Powell[1] and Martin and Moore[2] have shown that the hexagonal close-packed phase, which is found in beryllium at room temperature, transforms at approx 1250°C to a new phase which exists up to the melting point, approx 1280°C. The crystal structure of this latter phase has been determined by Martin and Moore[2] as bcc ($a_o = 2.55$A).

Since a bcc phase is inherently more ductile than a hexagonal close-packed phase (because of the many more slip systems available in the former), attempts have been made to stabilize the bcc phase to lower temperatures, possibly to room temperature.

The first attempts to attain this goal have been by means of alloying. A series of binary alloys of beryllium with iron, columbium, copper, vanadium, zirconium, palladium, silver, zinc, nickel, silicon, cerium, lanthanum, and chromium, and ternary alloys containing both nickel and palladium were investigated to determine the stability of the bcc phase in these alloy systems. The experiments are still in

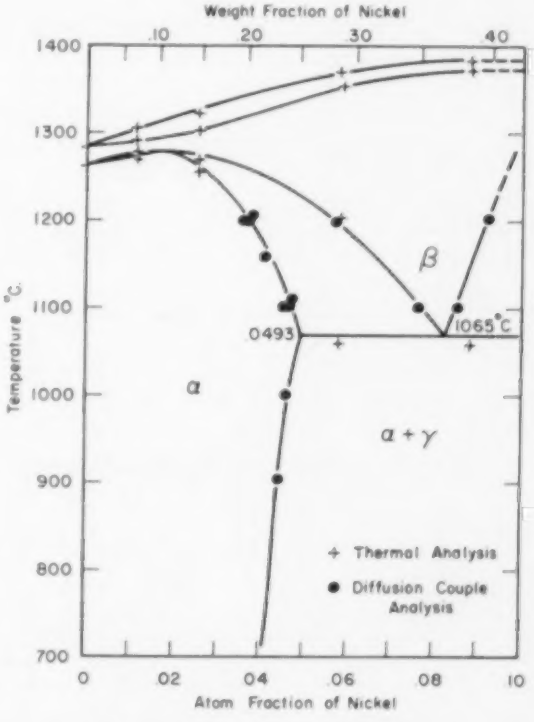


Fig. 1—Portion of the Be-Ni equilibrium diagram.

* Sponsored by the United States Atomic Energy Commission, Reactor Division, Contract No. AT-30-1-1565.

S. H. GELLES, J. J. PICKETT, and A. WOLFF are associated with Nuclear Metals, Inc., Concord, Mass. This paper was originally presented at the AIME Southwest Metals and Minerals Conference in Los Angeles, Calif., on April 22, 1960.

Table I. Chemical and Spectrographic Analyses of Starting Materials

Material	Chemical Analysis in ppm by Wt					Spectrographic Analysis in ppm by Wt									
	BeO	C	N	Cl	Fe	Al	Ca	Co	Cr	Cu	Fe	Mg	Mn	Ni	Si
Pechiney Be Flake	3100	155	30	700		~30	<50		<50	~35	~100	~10	<10	<30	<20
Pechiney Be Flake						30	<100		<40	40	160		<20	<20	10
Mond Ni Pellets		160-200			320-420			*							

* Undetected.

process; however, enough work has been done on the beryllium-nickel system to distinguish it as the most promising one investigated thus far.

The beryllium used in this investigation was in the form of flakes obtained from the Pechiney Co. of France; the nickel was in the form of pellets obtained from the International Nickel Co. Analyses of these materials are shown in Table I. The alloys were prepared by vacuum induction melting in beryllia crucibles.

Results

Fig. 1 shows the beryllium-rich portion of the beryllium-nickel phase diagram, based upon results obtained by diffusion-couple analysis, thermal analysis, and high-temperature X-ray diffraction. The alpha phase has the crystal structure normally found in pure beryllium at room temperature (hexagonal close-packed, c/a ~ 1.57). The beta phase is bcc, having a lattice parameter varying from approx 2.55A for pure beryllium[2] to 2.60A in the composition range 5 to 8 at pct Ni. The gamma phase, Ni_2Be_{21}, has a complex beta-brass structure, analyzed by Misch.[3]

The phase boundary points denoted in Fig. 1 by dots were obtained by a diffusion couple analysis technique described elsewhere.[4] The phase boundary points designated by crosses were obtained in this study by differential thermal analysis. Typical heating and cooling curves for a beryllium-nickel alloy containing 1 at pct Ni are shown in Fig. 2. These curves show the thermal effects on heating found during transformation from the alpha phase to the beta and during the melting of the beta phase. The reverse transformations are shown in the cooling curve.

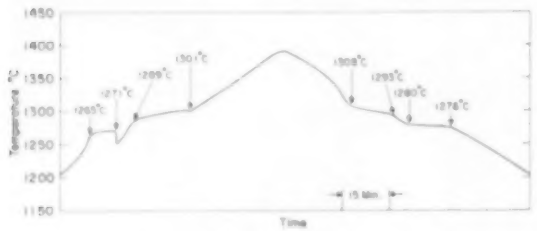


Fig. 2—Thermal analysis of Be-1 at pct Ni alloy.

Table II. Chemical and Spectrographic Analysis of Brush QMV Powder (—200 Mesh) used in Beryllium Aging Studies Wt Pct

	Brush Beryllium Co.	Nuclear Metals Inc.
BeO	0.88	0.62
Fe	0.158	0.159
Al	0.035	0.0335
Mn	0.011	0.0085
Ni	0.016	0.016
Cr	0.013	0.014
Co	0.0001	
Mg	0.015	0.016
Si	0.040	0.023
N		0.0299
C		0.036
Ca		<0.005
Cu		0.0008

Confirmation of the crystal structure of the beta phase by high-temperature X-ray diffraction was carried out with a vacuum furnace in conjunction with a Norelco Spectrometer. Three separate experimental runs were made on alloys containing 5 to 8 at pct Ni. A summary of the experimental conditions and results of these runs showed that the lines due to hexagonal beryllium and to Ni_2Be_{21} disappeared almost completely upon heating into the beta phase field. In the first two series of runs (A and B) involving Be-8 at pct Ni alloys, however, vestiges of Ni_2Be_{21} lines remained even at high temperature. This was found to be due to a change in composition of the sample during the experiment because of the evaporation of beryllium from the surface. As an example of this, the sample used in the series A experiments and analyzed after completion of the experiment was found to have an average composition of approximately double the nickel content of the starting material. A relatively thick layer of

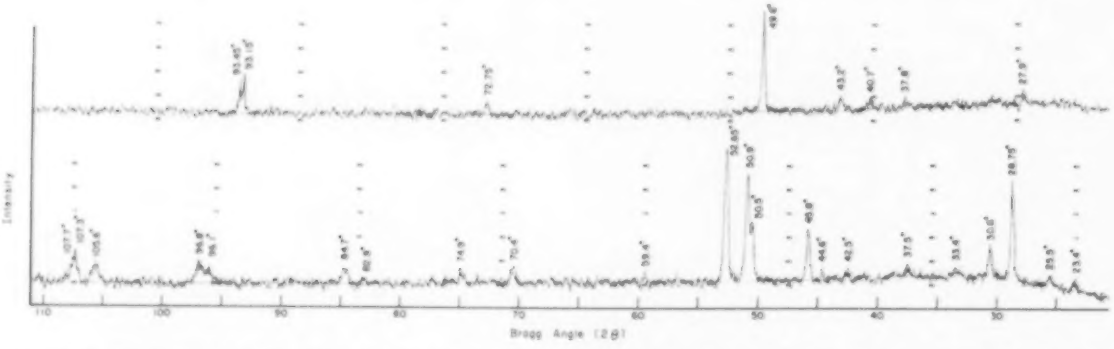


Fig. 3—Diffraction patterns of a Be-5 at pct Ni alloy at [a] room temp and [b] 1270°C. Scanned at 1° per min. CuKα radiation, 45 kv, 19 ma.

Ni_xBe_n would, thus, be expected to have formed on the sample surface. In an effort to avoid this problem, a hypoeutectoid alloy containing 5 at pct nickel was used in runs of Series C. The pattern obtained at 1270°C consists of three of the four bcc lines expected in the range of angles scanned. Only the slightest trace of a compound line is evident. This pattern is shown in Fig. 3, where it is compared to the pattern previously obtained at room temperature. It should be noted that the sample was at 1200°C or above for at least 20 min before the compound peak was scanned. This would allow enough time for the surface of the sample to become sufficiently enriched in nickel so that at least a thin layer of Ni_xBe_n would form at the surface of the sample.

Attempts have been made to stabilize the beta phase by quenching Be-8 at pct Ni alloys from approx 1200°C. Thus far, these attempts have been unsuccessful.

This work will be continued during the next year.

Beryllium Brittleness Problem

The second program to be described* is concerned with pinpointing the cause of brittleness in beryllium.

For many years now, workers in the field of beryllium metallurgy have blamed the brittleness of the metal either on the crystallographic structure of the pure metal (inherent brittleness)[5] or on impurities in the metal.[6]

The proponents of the inherent brittleness theory present theoretical arguments which are based on assumptions that have been necessarily simplified in order to solve the problem or are based on properties of the material in the impure state.

On the other hand, there are many experimental clues that lead to the belief that impurities in beryllium are affecting the mechanical properties of the metal. One of the prime reasons for this belief is the unusual temperature dependence of some of the mechanical properties of the metal. As an example, the tensile elongation as a function of temperature sometimes shows two maxima.[7] A decrease in elongation at a temperature of approximately 400°C can possibly be explained by formation of a precipitate and/or impurity-dislocation interactions.

In addition, the critical resolved shear stress for slip along the basal plane appears to increase with increasing temperature.[8,9] This is an effect which is difficult to interpret without assuming that impurities are active in beryllium.

By direct observation of fracture surfaces Pointu[10] has revealed the presence of impurity particles. These have been analyzed, with an electron microbeam probe, by investigators at Advanced Metals Research Corp. as being rich in iron, manganese, and chromium. Such particles, if concentrated along fracture planes, may have a deleterious effect on the mechanical properties.

As direct evidence of impurity effects, Mash[11] has demonstrated the presence of quench-aging and strain-aging effects in commercially pure beryllium. His data demonstrate that large changes in mechanical properties can be effected by heat treatment and cold work followed by heat treatment.

In the present work, some of Mash's conclusions regarding precipitation effects have beeen confirmed. In addition, the kinetics of an aging process in beryllium fabricated from commercial beryllium powder has been followed with electrical resistivity measurements and X-ray measurements. A limited number of tensile tests have been conducted on material fabricated from the same powder lot and treated in a similar manner.

Material

The beryllium rod used in this investigation was fabricated from −200 mesh Brush QMV powder. Chemical and spectrographic analyses of this material appear in Table II.

Experimental Procedure

Fabrication procedure: Beryllium rod approximately ½ in. in diam was fabricated from −200 mesh powder by extrusion at 1950°F at 30:1 area reduction. Two such extrusions were carried out; one was used to supply samples for resistivity tests and the other for tensile tests.

Aging kinetics studies: One of the extruded rods was cut into 4-in. lengths. These samples were solution treated at 1100°C for 1 hr and water quenched. They were then aged at temperatures between 200° and 1000°C for at least three different times at temperature. Fig. 4 shows the results obtained in a plot of the ratio of the resistivity of the aged sample to the resistivity of the solution-treated sample versus time. As may be seen from this figure,

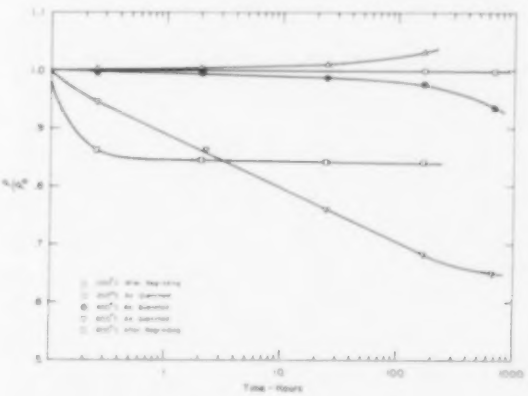


Fig. 4—Resistivity ratio (ratio of resistivity of aged specimen to resistivity of a as-solution-treated-and-quenched specimen) versus aging time for various aging temperatures.

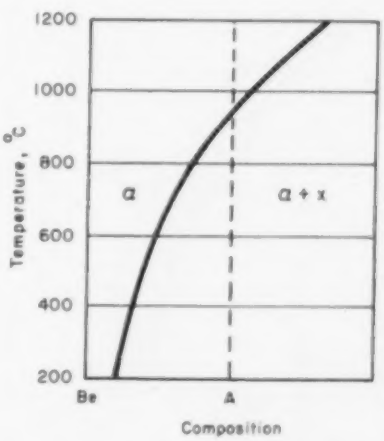


Fig. 5—Schematic phase diagram of beryllium with suspected solute impurity element. Drawn to be consistent with resistivity studies of aged samples of commercial beryllium.

* Sponsored by the United States Atomic Energy Commission, Reactor Division, Contract No. AT(30-1)-1565.

large changes in resistivity occur in commercial beryllium when subjected to heat treatment of the type described. This is especially true at the 600°C aging temp, where decreases of resistivity of the order of 30 pct are observed.

These changes in resistivity lead one to the belief that a precipitation process is taking place in commercially pure beryllium. The process occurs rather slowly in the temperature range 200° to 400°C. At 600°C it occurs rather quickly and at 800°C is complete in less than 2 hr. Aging at 1000°C produces only minor changes in the resistivity.

A possible explanation of these results can be made using the schematic diagram shown in Fig. 5. This diagram, which is drawn to be consistent with the resistivity results, shows the solubility curve of beryllium with an assumed solute element. The vertical line labelled "A" represents the composition of commercial beryllium used in this study. The alloy passes from a single- to a two-phase field at a temperature between 800° and 1000°C.

Work is now being carried out to identify the precipitated phase by means of the electron microbeam probe and by X-ray diffraction.

Changes in mechanical properties: The second extruded rod was cut into 3½ in. lengths and these samples were solution treated at 1100°C for one hour and either furnace cooled or water quenched. Some of the samples that were water quenched were aged at either 400° or 600°C for different lengths of time. They were machined into tensile bars of 1-in. gage length and 0.200-in. gage diam and tested on a Tinius Olsen hydraulic tensile machine at approximately 0.002 in. per min. A summary of the results is shown in Table III, where it is seen that the mechanical properties are altered quite substantially with heat treatment. Some of the significant factors to be noted are the following:

1) In comparing the samples slow-cooled from the solution temperature with those quenched from this temperature, significantly higher ultimate tensile strengths and higher elongations can be seen in the slow-cooled material.

2) Comparison of the samples quenched from the solution temperature with those quenched and aged at 400°C shows no significant change in either the ultimate tensile strength or the elongation even after 337 hr at this temperature. However, a yield point is developed in this material in as little as 2 hr at 400°C.

3) Aging at 600°C produces significant improvement in the ultimate strength and elongation over the solution treated and quenched properties. It is also significant that a yield point is developed in samples aged 24 hr at 600°C but is absent in samples aged 2 hr at 600°C.

4) The mechanical properties of the as-extruded metal, although based on a single sample, show a relatively high yield strength and elongation but a low ultimate tensile strength.

These results once again indicate that impurities are greatly affecting the mechanical properties of commercial beryllium. Inconsistencies and the spread of mechanical properties reported by investigators in the field of beryllium metallurgy are perhaps due to different thermal histories of the test samples. The precipitation process may also explain the decrease in ductility in the range 400° to 600°C. This is the range of temperature at which the precipitation process is beginning to take place at an appreciable rate. The embrittling mechanism appears to vary with temperature, perhaps being due to the presence of a fine precipitate in the temperature range 400° to 600°C and to impurities in solid solution at room temperature. This is evidenced by the increase in ultimate strength and elongation in room temperature tests on samples aged at 600°C.

An extensive investigation of this phenomenon is presently being carried out under Air Force sponsorship. From these studies we hope to learn what impurity or impurities are acting in beryllium and in what way they are altering mechanical properties.

References

[1] G. Powell: unpublished information, June 1956.
[2] A. J. Martin and A. Moore: The Structure of Beryllium with Particular Reference to Temperatures Above 1200°C, *J. of Less Common Metals*, vol. 1, no. 2, April 1959, p. 85.
[3] L. Misch: Structures of Intermetallic Compounds of Beryllium With Copper, Nickel, and Iron, *Zeitschrift fur physikalische Chemie*, vol. 29, 1935, p. 42.
[4] S. H. Gelles, R. E. Ogilvie, and A. R. Kaufmann: The Solid Solubilities of Iron and Nickel in Beryllium, *AIME* Trans., vol. 215, no. 4, Aug. 1959, pp. 695-702.
[5] S. F. Pugh: Relations Between the Elastic Moduli and Plastic Properties of Polycrystalline Pure Metals, *Phil. Mag.* ser. 7, vol. 45, no. 368, 1954, p. 823.
[6] W. Kroll: Is Beryllium Ductile?, *Metals and Alloys*, vol. 8, 1937, p. 349.
[7] A. R. Kaufmann, P. Gordon, and D. Lillie: The Metallurgy of Beryllium, *ASM Trans.*, vol. 42, 1950, p. 785.
[8] H. T. Lee and R. M. Brick: Deformation of Beryllium Single Crystals at 25° to 500°C, Final Report on ONR-24908, University of Pennsylvania, Sept. 1953.
[9] G. L. Tuer and A. R. Kaufmann: Ductility of Beryllium as Related to Single Crystal Deformation and Fracture, *The Metal Beryllium*, ASM, Cleveland, Ohio, 1955.
[10] P. Pointu: personal communication.
[11] D. R. Mash: Aging Effects in Commercially Pure Beryllium, JOURNAL OF METALS, vol. 7, 1955, p. 1235.

Table III. Mechanical Properties of Beryllium Rod Extruded from Brush QMV Powder As a Function of Heat Treatment

(Tested at room temperature. Four samples tested for each condition unless otherwise specified.)
Solution treated at 1100°C for 1 hr

Heat Treatment	0.2 Pct Offset Yield Strength (psi)			Yield Point (psi)			Ultimate Tensile Strength (psi)			Elongation Pct		
	Max	Avg	Min	Max	Avg	Min	Max	Avg	Min	Max	Avg	Min
Water quench[a]	41,500	41,200	40,250	No yield point noted			68,900	61,640	60,600	3.5	2.5	1.5
Slow cool	41,600	40,925	40,200	No yield point noted			89,300	86,090	79,900	18.7	12.4	6.7
Water quench, 2 hr at 400°C[b]	44,000	44,000	44,000	44,000	43,890	43,700	68,200	58,260	52,700	3.5	2.1	1.4
Water quench, 24 hr at 400°C[b]	44,500	44,000	43,250	44,300	43,800	42,900	64,700	59,950	52,800	2.9	2.3	1.5
Water quench, 337 hr at 400°C	42,400	42,180	41,900	42,400	41,750	41,000	62,600	59,320	55,600	2.6	2.2	1.6
Water quench, 2 hr at 600°C	40,300	40,200	40,000	No yield point noted			74,200	66,270	60,400	4.6	3.2	2.1
Water quench, 24 hr at 600°C	46,000	45,325	44,200	46,700	45,320	44,200	88,600	84,530	80,400	18.0	11.6	6.6
As-extruded[c]		Not measured			50,000*			58,000			17.6	

[a] Five samples tested.
[b] Three samples tested.
[c] One sample tested; no solution treatment.
* Repeated yielding noted.

NUCLEAR PURITY BERYLLIUM . . . Its Manufacture

The valuable properties of beryllium in nuclear devices and reactors are nullified by high-neutron-absorption impurities. This article describes techniques for refining beryllium to a purity suitable for nuclear applications.

by C. W. Schwenzfeier, Jr.

The usefulness of beryllium in nuclear devices and reactors is based on three properties: its ability to moderate or slow down thermal neutrons, its ability to reflect thermal neutrons, and its low neutron capture cross section. These valuable properties of beryllium, however, are nullified if the metal contains an excessive amount of high-neutron-absorption impurities. For this reason, the metal must be produced to very high standards of purity. Another reason for requiring high-purity beryllium in nuclear applications is that certain elements, after exposure to irradiation in a nuclear reactor, produce appreciable quantities of highly-radioactive, long half-life isotopes. If the beryllium contains any appreciable quantity of these elements, of which cobalt is an example, the beryllium will remain radioactively hot for long periods of time and, thus, will be awkward and difficult to remove from a reactor. This could be a serious problem in such applications as beryllium cladding for fuel elements.

The double and unrelated requirements of high-purity, low-neutron absorption and avoidance of long half-life highly-radioactive isotopes, have given rise to a chemical purity specification for beryllium which is not a requirement for metals outside of the nuclear reactor field. The result of this situation is a double chemical specification, one part of which is based upon the sum of impurity elements properly weighted to reflect their respective neutron-absorption characteristics, and a second specification setting absolute maximum-allowable levels of the elements producing highly-radioactive long half-life isotopes. There is also some correlation between mechanical properties of beryllium and certain impurity elements, and this has resulted in maximum allowable levels for certain other impurity elements. For over 10 years, the beryllium industry has been producing nuclear-grade beryllium metal to the dual specification established by the US Atomic Energy Commission. This dual specification and its details are too complex to discuss here; however, an analysis of a typical lot of metal produced to this specification is presented in Table I.

Beryllium metal of a quality corresponding to this specification—commonly referred to as QMV*—is the standard for use both in nuclear and, more recently, in aircraft and missile applications. Beryllium metal of this nuclear quality was first produced in quantity during the early 1940's at the Lorain, Ohio, plant of The Brush Beryllium Co. in connection with the Manhattan Project.

Increasing demand for nuclear-grade beryllium resulted in the construction of a new beryllium plant at Luckey, Ohio, which went into operation in 1950. This plant, operated by The Brush Beryllium Co. for the US Atomic Energy Commission, remained the sole source of production quantities of nuclear-grade beryllium until approximately two years ago when, again, sharply rising demands for the metal resulted in the construction of two new privately-owned plants: one operated by The Beryllium Corp. and constructed at Hazelton, Pa., and the other constructed and operated by The Brush Beryllium Co. at Elmore, Ohio. While there has been some increase in quantity of nuclear-grade beryllium production in France, the two privately-owned US plants are today the only truly production-scale beryllium manufacturing operations in the free world.

Production steps

Production of beryllium metal, whether of nuclear quality or otherwise, is accomplished basically in two fundamental steps. The first of these is extraction of beryllium from its ore, beryl, a beryllium aluminum silicate, and the conversion of the extracted beryllium into either beryllium hydroxide or beryllium oxide. The bulk of the impurities associated with beryllium in its ore are separated in the course of producing the oxide or hydroxide.

The second basic step in beryllium metal production is conversion of the oxide or hydroxide to either the anhydrous fluoride or the anhydrous chloride, followed by either reduction of the fluoride with magnesium metal or electrolytic reduction of the anhydrous chloride. Final purification to meet the nuclear specifications is accomplished in the halide portion of the processing operations. In the case of the fluorides, this final purification is accomplished primarily by wet chemical methods. Final purification of the chlorides may be accomplished by either distillation of the chloride or discarding the initial electrolytic beryllium deposit, which is high in impurity elements.

Insofar as chemical purity of the final beryllium product is concerned, it now appears that there is little difference between the fluoride-thermal reduction process and the beryllium-chloride-elec-

* Trademark of The Brush Beryllium Co.

C. W. SCHWENZFEIER, JR., is vice president of Brush Beryllium Co. This paper was presented at the 1960 AIME Annual Meeting.

trolysis process. However, as recently as a year ago, the chloride-electrolysis process was considered inherently superior to the fluoride-thermal reduction process.

Early in 1959, the UK Atomic Energy Authority approached the Brush Beryllium Co. with a request for assistance in obtaining larger quantities of beryllium metal than had been previously supplied for use in the British gas-cooled power reactor program. They had been using some French flake metal, but most of their beryllium came from the US, produced to standard US Atomic Energy Commission specifications. Comparing the results of their experiments using both standard large-scale fluoride-thermal reduction process beryllium from the US and European-produced laboratory scale chloride-electrolysis process metal, it was felt at that time, that worthwhile improvements in nuclear reactor design and/or fuel recovery could be realized through the use of beryllium of considerably higher purity than the standard US A.E.C. grade. The inadequate production capacity in Europe and the substantially higher cost of electrolytic process beryllium encouraged the British to consider a solution to the problem.

A comparison of the typical analysis of beryllium metal produced according to the US A.E.C. specification and the purity specification required by the UK Atomic Energy Authority is shown in Table II. A crash program of process development and refinement was started immediately at the Elmore plant of The Brush Beryllium Co. Within a few months, process modifications and improvements were made in the main plant so that for the first time beryllium metal of a chemical purity previously thought possible only from the chloride-electrolysis process became available in large quantities at only a small extra cost.

Table III shows an analysis of a typical lot of this new special purity grade of beryllium. In recognition of its current availability in quantity production, this new grade of beryllium metal has been given the trade designation of QMV N-50-A.

Table I. Typical Analysis of Beryllium Metal Produced to USAEC Specification

	ppm
Al	600
B	1
Cd	0.7
C	600
Co	1
Fe	1100
Li	1
Mg	300
Mn	80
Ni	120
Si	300

Table II. Typical Analyses of Beryllium Metal Produced for USAEC and UKAEA

	USAEC, ppm	UKAEA, ppm
Al	600	500
B	1	0.8
Cd	0.7	0.7
C	600	500
Co	1	1
Fe	1100	450
Li	1	1
Mg	300	150
Mn	80	50
Ni	120	110
Si	300	125

Table III. Specifications for UKAEA and Typical Analyses of Metal Made to Brush Specification N-50-A

	UKAEA Specification, ppm	Analyses To N-50-A (Lot #413), ppm
Al	500	400
B	2	1
Cd	2	0.7
C	700	400
Co	5	1
Fe	500	360
Li	3	1
Mg	150	90
Mn	50	50
Ni	100	95
Si	300	45

Ore extraction

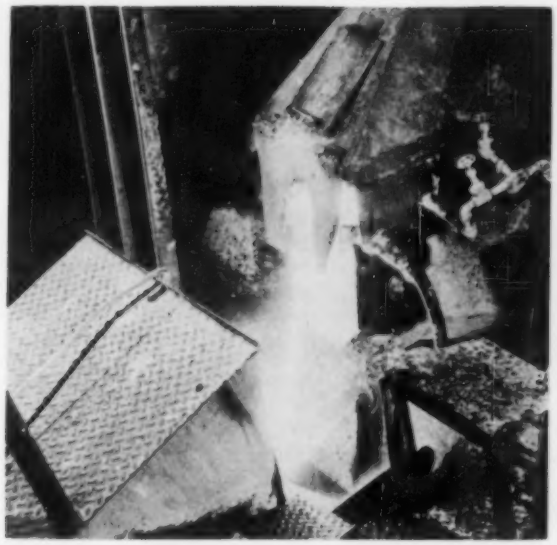
As stated earlier, production of metallic beryllium can be considered in two basic steps: production of beryllium oxide or hydroxide from the ore, and conversion of the oxide or hydroxide to a halide, with subsequent reduction of the halide to metal. Of the many ore extraction processes used commercially over the years, only two remain in operation in this Country on a commercial scale. No information is available to indicate what ore extraction methods may be in use outside of this country.

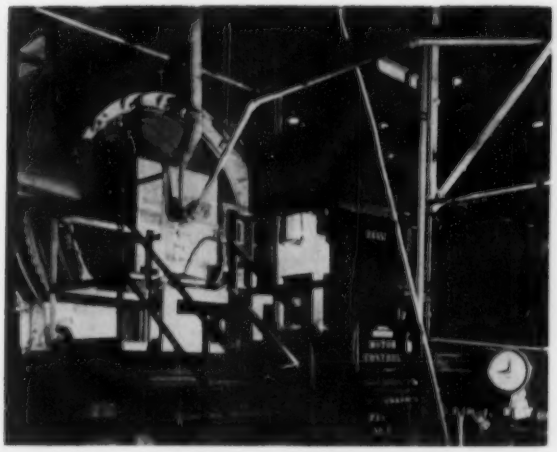
At its beryllium plant at Hazelton, Pa., The Beryllium Corp. prepares their beryllium oxide by the alkali-metal double-fluoride sintering process. Basically, this process consists of three steps: sintering at approx 750°C briquettes of a finely-divided mixture of beryl ore and an alkali metal double-fluoride, such as sodium ferric fluoride or sodium silico fluoride; leaching the ground sinter cake with water to recover the beryllium as water-soluble sodium beryllium fluoride, and then precipitating beryllium hydroxide from this solution by addition of sodium hydroxide. No further information on processing technology at Hazelton has been released.

In brief, the process employed by the Brush Beryllium Co. at its Elmore, Ohio, plant consists of melting the beryl ore, water-quenching the melt, reacting the ground frit with sulfuric acid to convert the beryllium to water-soluble beryllium sulfate, separating aluminum by crystallization of ammonium aluminum sulfate, and precipitating the beryllium as beryllium hydroxide with sodium hydroxide. The undesirable heavy metal impurities are retained in solution in the precipitation step through the use of suitable sequestering agents.

At Elmore, the beryl ore is melted in a 1500-kw carbon-lined, three-phase arc furnace of the type used in electric-furnace steel practice. Molten ore is poured from the furnace into water to achieve a quick quench, thus destroying the crystal form of the mineral, beryl, so as to make it reactive with sulfuric acid. No flux additions of any sort are made in this melting operation. (Fig. 1)

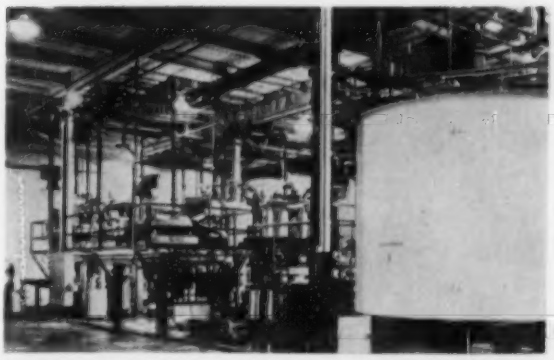
Following the quenching operation, the beryl frit is heat treated in a gas-fired rotary kiln to further improve reactivity. The heat-treated frit is ball-milled dry to pass a 200 mesh sieve and then mixed with 66° Baume sulfuric acid. From the ore-acid mixer, the slurry is blown by air pressure into preheated, gas-fired sulfating reactors, similar in construction to rod-mill type pulverizers. The sulfating reaction temperature is approx 300°C. In the sulfating reaction, the beryllium oxide content of the ore is converted into water-soluble beryllium sulfate, and the silica content of the ore is completely dehydrated to make it insoluble. (Fig. 2)

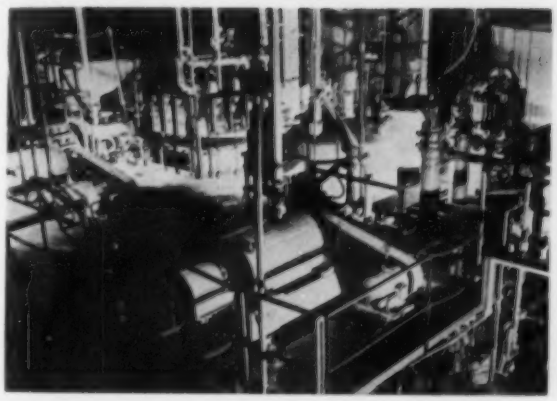




STEP ONE, EXTRACTION

Fig. 1 (left)—Fritting of molten beryl ore. Fig. 2 (above)—Feed end of sulfating mill. Fig. 3 (below)—Continuous centrifuges and thickeners. Fig. 4 (bottom)—Alum centrifuge crystallizer.





Following sulfating, the ore is water-leached in a five-stage countercurrent Dorr thickener. The silica sludge from the fifth thickener stage is virtually 100 pct free of water-soluble beryllium and is discarded. The thickener operation is, of course, continuous and fully automatic. Fully automated processing of the purified supernatant liquor from the No. 1 thickener stage continues with the addition of ammonium hydroxide, and the cooling of the solution in tube and shell type heat exchangers to cause separation of the aluminum as sparsely-soluble ammonium aluminum sulfate, which is removed continuously in a Bird solid-bowl centrifugal. The alum crystals are washed free of beryllium in the centrifuge prior to discard, and the wash water is combined with primary centrifuge filtrate. A small amount of extremely-fine silica remains in the supernatant liquor from the first thickener stage; so, at this point, after alum crystallization, the filtrate is given a polishing filtration in tank-type pressure filters, where the small amount of extremely fine alum not removed by the Bird centrifuge, as well as the residual silica fines are separated from the liquor and discarded. The clarified filtrate is next blended with suitable organic chelating agents for sequestering of undesirable heavy-metal impurities. Sodium hydroxide solution is simultaneously proportioned into the stream in proper amount to produce a final blend approximately 1.5 N basic. Under these conditions, heavy metal impurities are held in solution by the sequestering agents, the beryllium content of the solution has been converted to sodium beryllate, which is water soluble at temperatures below approx 60°C, and the residual aluminum content has been converted to water-soluble sodium aluminate. (Figs. 3 and 4)

The clear solution is transferred continuously to a steam-heated hydrolyzer, where the body of the solution is held at approx 95°C, thus reprecipitating the beryllium hydroxide in the very coarse, granular alpha form. From the hydrolyzer, the alpha beryllium hydroxide slurry flows continuously to another Bird solid-bowl centrifuge, where the solid hydroxide is separated from the basic mother liquor. Under normal operating conditions, approx 95 pct of the solid beryllium hydroxide is separated in the centrifuge and the remaining 5 pct must be recovered by settling in a Dorr thickener. The clarified beryllium-free basic mother liquor is discarded along with the ammonium alum crystals. By fortunate circumstance, the amount of excess caustic soda in this clarified filtrate is almost exactly equivalent to the amount of aluminum present in the ammonium alum produced; so, when these two waste streams are blended in the waste treatment ponds, all of the aluminum is precipitated, and the resultant supernatant liquor is almost exactly neutral.

Typical beryllium hydroxide produced by this process contains only about 10 pct free water and,

on a BeO basis, approx 0.05 pct Fe, 0.15 pct Al, 0.05 pct Si, and 0.45 pct Na.

This completes the first basic step of beryllium metal production—extraction of beryllium from the ore and its conversion to beryllium hydroxide.

To a fluoride

The second basic step of metal manufacture—conversion of the hydroxide to anhydrous fluoride and thermal reduction of the anhydrous fluoride to metal—begins with preparation of a solution of ammonium beryllium fluoride by dissolving the hydroxide in a solution of ammonium acid fluoride. Hydroxide powder from the centrifuge falls directly into an agitated tank where it is slurried with an ammonium bifluoride solution and transferred to large agitated tanks to be stored in slurry form. This slurry is withdrawn from the storage tanks as needed and transferred to agitated Haveg tanks, where additional hydrofluoric acid and ammonium hydroxide are added to balance properly the stoichiometric relationship of beryllium, ammonium, and fluoride ions in the solution.

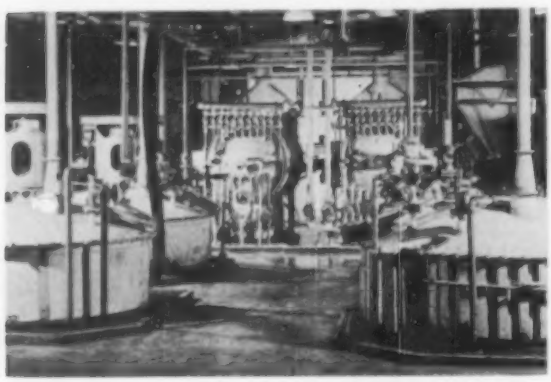
Several years ago, we discovered that when these stoichiometric relationships are properly balanced, a complex is formed that dissociates to such a slight extent that the quantity of beryllium ions in the solution is so small as to preclude exceeding the solubility product of beryllium hydroxide. This complex ion is the key to the basic beryllium purification process, since it makes possible the setting of solution pH to any value desired to accomplish precipitation of the heavy metals present without the danger of losing beryllium, as beryllium hydroxide precipitate, in the filter cake.

In the first purification step, the solution is adjusted to a pH of approx 7 and lead peroxide is added to precipitate, practically quantitatively, manganese as manganese dioxide and chromium as lead chromate. The precipitate is separated by filtration. The clarified filtrate is heated to boiling in an agitated Haveg tank, and then calcium carbonate is added to precipitate residual aluminum. Under these conditions, there is some decomposition of the ammonium fluoride present in the liquor to release ammonia, which raises the pH of the solution to approx 8.5. At this pH, the hydroxides of most of the heavy metals are precipitated quantitatively. The hydroxides of some metals, however, are not quite sufficiently insoluble to assure adequate solution purity; therefore, ammonium sulfide is added to assure complete separation of copper, nickel, and ferrous iron. This solution is filtered; the filter cake is thoroughly washed and is then discarded.

To eliminate any possible water pollution problem resulting from the fluorides contained in these discarded sludges, they are mixed with the calcium hydroxide sludges generated in the main refinery water-treating plant. Additional lime may be added, if needed, to assure conversion of all fluorides to highly insoluble calcium fluoride.

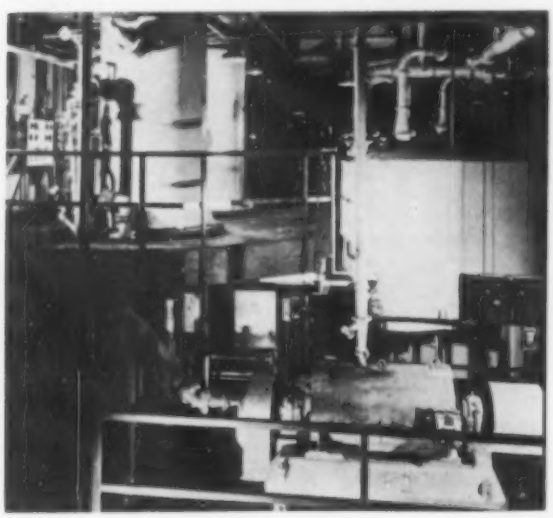
To make doubly sure that there will be no quality problems arising in subsequent processing operations, the clarified ammonium beryllium fluoride solution is filtered again through fine filter papers in tank-type pressure filters. (Fig. 5)

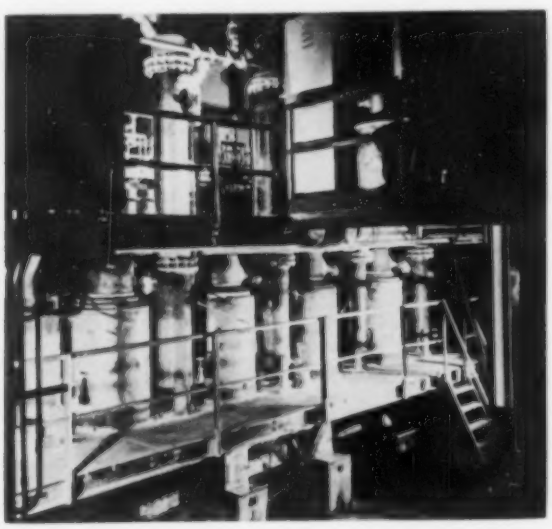
The next step in the process is evaporation and crystallization to produce the salt; ammonium beryllium fluoride. This operation is carried out in a continuous, automatic, vacuum salting evaporator. Crystallization of the salt is affected by the stoichiometric balance of ammonium, beryllium, and fluoride ions; therefore, the clarified filtrate must be checked for ammonium fluoride balance prior to evaporation and crystallization. In the calcium car-



HYDROXIDE TO FLUORIDE

Fig. 5 (above)—Pressure filters in ammonium beryllium fluoride treating. Fig. 6 (below)—Vacuum salting evaporator and continuous centrifuge for ammonium beryllium fluoride. Fig. 7 (right)—Induction furnaces for producing beryllium fluoride.





bonate treating operation, some ammonium fluoride is lost, and this loss must be replaced at this point in the process. Ammonium fluoride is added in the form of a solution that has been processed through the same purification operations as the main liquor stream. The evaporator is constructed of rubber-lined steel, with all process piping either polyvinyl-chloride or Haveg. The external heat exchanger is of graphite construction. These precautions in equipment construction are necessary in order to eliminate any possibility of recontamination of the process liquors. The evaporator operates at a temperature of approx 50°C and is fully automated. Salt crystals produced in the evaporator body are removed continuously by a Bird solid bowl centrifuge. The salt-free filtrate is returned continuously to the evaporator body, with a small mother liquor bleed-off. The ammonium beryllium fluoride salt feeds continuously to a drier, and the dry salt is conveyed to an epoxy-lined storage hopper. (Fig. 6)

In the next processing operation, the ammonium beryllium fluoride salt is decomposed thermally into its two component parts: ammonium fluoride and beryllium fluoride. This reaction is carried out in induction-heated furnaces, with all furnace parts that come in contact with the salt or beryllium fluoride constructed of graphite. These furnaces are charged continuously by feed conveyors from the storage hopper. At the furnace operating temperature of 900°C, the ammonium fluoride is driven off as a gas and absorbed in water. The gas absorber consists of a packed tower in series with a water-flushed electrostatic collector. The ammonium fluoride solution recovered in this operation is acidified with hydrofluoric acid and returned to the hydroxide dissolving step at the head of the process. (Fig. 7)

The beryllium fluoride flows continuously as a liquid from the bottom of the furnace crucible through a bubble-cap-type gas seal. This fluoride stream falls onto water-cooled casting wheels where the beryllium fluoride solidifies in the form of roughly-hemispherical chunks about the size of a tennis ball.

The beryllium fluoride product from the five decomposition furnaces is conveyed continuously to an automatic batch weighing station. At this station, the beryllium fluoride lump is discharged into stainless steel charge containers. When the charge container has received an amount equal to one reduction furnace batch charge, it is automatically removed and replaced by an empty container.

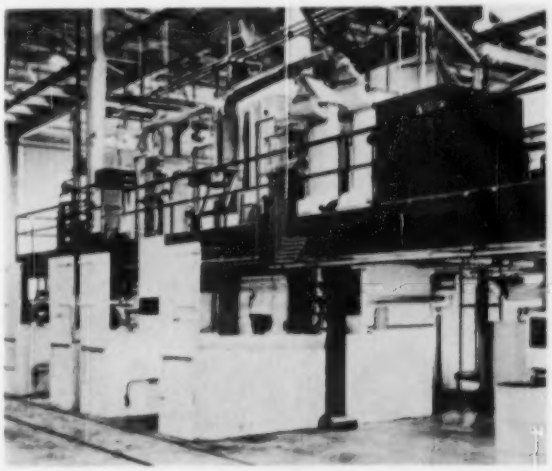
Fluoride to metal

The beryllium fluoride is reduced to beryllium metal in induction furnaces provided with graphite crucibles. The reducing agent is high-purity magnesium metal, which is charged to the furnace in lump form, the lumps being approx 1-in. cubes. Although the reduction reaction takes place a little below 1000°C, the entire charge must be heated to nearly 1300°C to permit coalescence of the beryllium into pieces large enough to recover in subsequent operations. In order to assure smoothness of reaction and also to simplify metal-slag separation later on, excess beryllium fluoride over that stoichiometrically equivalent to the magnesium is used in this reduction reaction. The amount of magnesium charged is stoichiometrically equivalent to only about 75 pct of the beryllium fluoride charge. (Fig. 8)

Metal and slag are poured together from the reduction furnace into graphite receiving pots. After the metal-slag mass has solidified, it is crushed in a hammer mill and then water leached in a ball mill. During milling, a continuous flow of leach water is pumped through the ball mill and out into settling tanks. This water stream flushes the more friable magnesium fluoride slag out of the mill during grinding, finally leaving just the slag-free beryllium lumps and steel grinding balls in the mill. The ball mill load is passed over a magnetic separator to remove the grinding balls, which are recycled to the next ball mill charge. The beryllium pebbles, after magnetic separation, are ball milled again in a smaller mill with dilute acetic acid for final slag removal and clean-up.

Final operation

The final operation in the process for producing nuclear-grade vacuum-cast billet is vacuum casting. The vacuum casting furnaces have a beryllium metal capacity of approx 75 to 80 lb. The melting crucible is made of beryllium oxide, and the charge is heated directly by induction. The purpose of this operation is mainly to remove residual magnesium metal and slag. The average assay of pebbles charged to the vacuum casting operation is 96.5 pct Be. The finished castings, which are poured in graphite molds, will assay in excess of 99.5 pct Be. The magnesium content of the pebbles charged to the furnace may be as high as 2 pct, while the magnesium content of the finished casting will not exceed a few hundred ppm. (Fig. 9)



REDUCTION

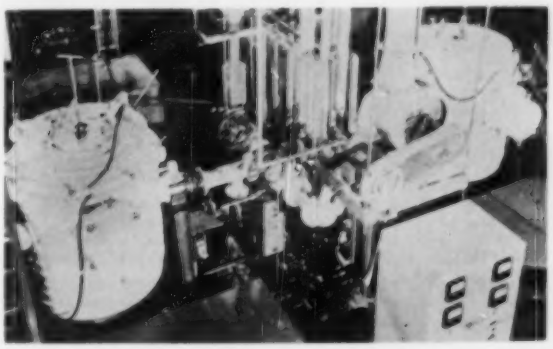


Fig. 8 (above)—Induction furnaces for reduction of beryllium fluoride with magnesium. Fig. 9 (below)—Vacuum casting furnaces.

HIGHER PURITY INGOT CERIUM FROM MOLTEN SALTS

Based on the experimental results obtained at the Reno Metallurgy Research Center of the US Bureau of Mines, an evaluation of several critical factors in the electrowinning of cerium from molten salts is presented.

by B. Porter, E. S. Shedd, C. Wyche, J. D. Marchant and R. G. Knickerbocker

The marked interest being shown in molten-salt systems is amply demonstrated by the large number of papers in recent scientific literature. Many references pertinent to electrolytic production of rare-earth metals have already been presented by Morrice[1, 2] in two reviews.

In many ways the electrowinning of metals from molten salts is still an art rather than a science. Most of the data available for work in aqueous mediums—electrical resistivities, solubilities, etc.—have to be measured for each molten-salt system, since they are unknown. The need for this basic research has been of much concern to the scientific world. A recent editorial in the June 1959 issue of the AIME JOURNAL OF METALS states the case very well.[3]

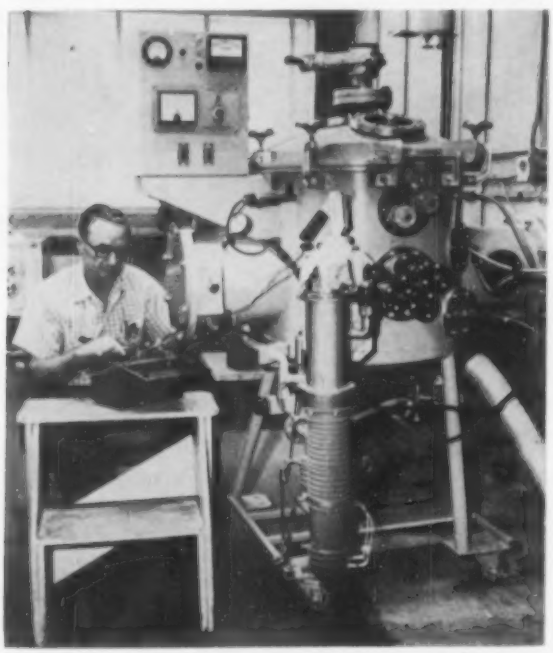


Fig. 1—Controlled atmosphere-temperature-pressure (CATP) capsule. Present temperature operating range, room to 1600°C.

B. PORTER, E. S. SHEDD, C. WYCHE, J. D. MARCHANT, and R. G. KNICKERBOCKER are with the Reno Metallurgy Research Center, US Bureau of Mines, Reno, Nev. This paper was presented at the 1960 AIME Annual Meeting.

The Reduction and Refining Research Group of the Reno Metallurgy Research Center of the US Bureau of Mines is engaged in preparing higher purity rare-earth metals. Table I presents the analysis of one typical sample of cerium metal prepared at Reno.

This paper reports some of the basic work being done concurrently to help place this laboratory procedure on a sounder scientific footing.

Apparatus

A) **Cerium Electrowinning Unit:** The electrowinning cell and the controlled atmosphere box currently in use were described previously[4]. In brief, the cell consists of a molybdenum cathode, carbon anode, a suitable graphite container, and a molten bath prepared from a mixture of 12 wt-pct Ba, 15 pct Li, and 73 pct cerium fluorides. The entire cell is inside a steel gloved box in which the requisite inert atmosphere can be maintained. Services are introduced through vacuum-tight fittings, and necessary manipulations can be made through gloved ports.

B) **Controlled Atmosphere - Temperature - Pressure (CATP) Capsule:** A CATP capsule intended for use in smaller scale investigations was designed and built, as shown in Fig. 1. It consists of a steel cylindrical work chamber that is 24 in. in diam and 34 in. in height and has a steel plate welded across the bottom. The top closure is made by means of a plexiglas hemispherical dome, or a steel one, that seals against an O-ring. The CATP capsule is provided with gloved ports, vacuum valves for gas sampling, thermocouple lead—throughs, and power leads for resistance and induction heating. Evacuation of the chamber is achieved by a combination of mechanical and diffusion pumps.

Much of the work reported here was done in this capsule, which has proved satisfactory for temperatures that were as high as 1600°C inside the cells used for the experimental investigations. The remainder of the experimental work was carried out in the cerium electrowinning unit.

C) **Equipment for Measurement and Control of the Atmosphere:** Moisture content of the cell gas is measured by a moisture monitor that can detect as little as 1 ppm of water in a gaseous atmosphere. Oxygen, carbon dioxide, and carbon monoxide are measured with precision gas-analysis equipment

that gives a minimum reading of 0.1 pct. To afford a quicker analysis of the oxygen content of the working atmosphere of a cell in operation, a Kwik-Chek calibrated for a minimum reading of 0.25 pct is employed. To allow an overall test of the quality of the inert atmosphere in the gloved box, the thermal conductivity of this gas is compared with that of pure reference gas by using a gas master.

A larger cell is necessary if a product that will lend itself to significant tests is to be obtained. A large, single ingot, once trimmed, would presumably be more homogeneous than those prepared in small-scale laboratory experiments. Such a cell is being constructed at the Reno Metallurgy Research Center.

Experimental

A) **Oxygen Gas Found in the Cell Atmosphere:** During some of the electrolyses to produce cerium metal from molten salts, routine monitoring of the gloved-box atmosphere has shown the presence of oxygen gas in small amounts. Before operation of the cell is begun, the box atmosphere is changed enough times so that a zero content of oxygen, carbon monoxide, and carbon dioxide is indicated on the instruments previously described. As the chamber then is ready for use with inert atmosphere, either helium or argon, the direct-current source is activated.

A series of experiments has been conducted to locate the source of this oxygen as follows:

1) A cold Vycor container was placed in a helium atmosphere inside the gloved box and vacuum-dried cerium dioxide was fed into it.

2) Experiment 1 was repeated, using a cold graphite container in place of the inert one. It was again repeated, with the graphite container held at a temperature of 835°C. Cold, vacuum-dried cerium dioxide was fed both by hand and by means of a vibra-screw mechanism used to feed the electrowinning cells currently under study.

3) The undried cerium dioxide was heated in a graphite container at temperatures over 800°C in an inert atmosphere.

4) The vacuum-dried cerium dioxide was fed into the molten electrolyte used to produce cerium metal, which was contained in a graphite crucible. The atmosphere was argon. Electrolysis was not attempted in this experiment.

5) Experiment 4 was repeated, followed by electrolysis of the melt to produce cerium metal.

Only in some of the experiments of type 5 was molecular oxygen detected. In experiment types 2, 3, and 4, small amounts of carbon monoxide and dioxide were detected. Samples of the gaseous atmosphere, in which cerium was produced, were sent for analysis by mass spectrometer. The first results showed that the ratio of nitrogen to oxygen in these gas samples is not that of air. The analysis of a recent gas sample confirmed the original results—Table II.

B) **Valence of Cerium Reduced in the Electrowinning Experiments:** Table III presents some data obtained in one typical electrowinning experiment.

It is entirely possible that neither ion valence is the only one reduced. An easy calculation, based on the assumption that the metal produced in this run is equivalent to 100-pct current efficiency, indicated that the effective valence of the species reduced to form the cerium metal is approx 3.6. This was determined in spite of the fact that enough quadrivalent ion, in the form of cerium dioxide, was introduced into the bath during the run to account for production of 790 g of cerium metal.

In another run cerium dioxide was not added to the cell bath, but cerium metal was produced continuously (at a very poor current efficiency), establishing the fact that reduction of trivalent cerium, introduced into the original bath in the form of CeF_3, is possible.

Experiments now in progress are expected to furnish the data required to decide whether trivalent cerium ions alone, or a combination of tri- and

Table I. Analysis of One Typical Sample of Cerium Metal Prepared at Reno

Elements	Weight Pct[a]
Fe	0.02
Si	[c]
Al	<0.01
Ca + Mg	<0.004
Li	0.002
Ba	[c]
Mo	[c]
Cr	[c]
Mn	[c]
Total rare earths other than cerium	[c]
C[b]	0.016
O[b]	0.036
H[b]	0.0003
N[b]	[d]

[a] Spectrochemical analysis.
[b] Vacuum fusion analysis.
[c] Not detected.
[d] Not analyzed.

Table II. Mass Spectrometric Analysis of Gas Taken From Area Above the Cell During Electrowinning in CATP Capsule

	Mol, Pct	
Identity	Sample A[a]	Sample B[b]
H	0.04	0.06
CO	—	0.42
N	0.07	0.20
O	0.02	0.31
Ar	99.87	89.63
CO_2	—	9.35
CF_4[c]	—	0.03
	100.00	100.00
H_2O	(0.10)	(0.18)

[a] Bath molten, instruments show absence of impurities prior to start of electrolysis.
[b] During electrolysis, note N/O ratio less than unity.
[c] Tentatively identified only.

Table III. Data Obtained in One Typical Electrowinning Experiment for Preparation of Higher Purity Ingot Cerium Metal

Weighted avg amperage (dc)	250 amp
Time of electrolysis	132 min
Cerium metal recovered	797 g
Cerium dioxide fed	971 g

From these data the following has been calculated:

Faradays used in this experiment	20.52
Theoretical yield based on reduction of only quadrivalent ions[a]	718 g
Theoretical yield based on reduction of only trivalent ions[a]	958 g
Pct theoretical yield (quadrivalent basis)	111
Pct theoretical yield (trivalent basis)	83

[a] Not intended to imply that this is the actual form of the ion present in the melt.

quadrivalent ions, are reduced to cerium metal in the electrowinning cell.

C) **Nodule Formation:** This phase of the project is one of the most important. From the 56 electrowinning runs made at Reno, the following outlined trends have become apparent:

1) Nodule formation, as opposed to formation of one large ingot, appears to take place: a) when oxide feed into the bath is intermittent, and b) when the electrolytic current is interrupted.

2) Use of a *skull* technique (that is, formation of a layer of frozen bath on the sides and bottom of the graphite container to prevent cerium from contacting it) has resulted in large, single ingot formation in some experiments. In other experiments, it had little effect, and the product was a multitude of small nodules.

D) **Analysis of Cerium:** The analysis of reactive, high-purity metals has been the subject of much continuing effort. Both metallic and non-metallic impurity concentrations have been shown to be important in evaluating the quality of the metal produced. Several methods of attack being employed by the Reno Metallurgy Research Center are listed:

1) Spectrochemical determination: The sensitivity of methods now available does not seem adequate for the analysis of high-purity metal, since in many instances values must be quoted at less than a figure in the second decimal place.

2) Nonmetallic impurities (Carbon, sulfur, oxygen, hydrogen, and nitrogen, in whatever form they may take in the metal): At present, only carbon and sulfur have been determined at the Reno Metallurgy Research Center. Because standards were lacking, these analyses were based on samples of iron certified by the National Bureau of Standards. Burning the cerium metal sample in a crucible containing an iron accelerator was expected to provide enough similarity in matrix to allow a sensible accuracy in this determination. Precision of the method is of the order of ± 0.003 pct absolute. The sulfur content of all analyzed Reno electrocerium was of the order of <0.002 pct, and the carbon content ranged from 0.005 pct to as high as 0.2 pct. One major complication in these analyses was the lack of homogeneity of samples produced in a single electrolytic run.

3) Neutron activation analysis: Samples were submitted to Oak Ridge for analysis. Some of the difficulties involved in analyzing cerium by this method are being investigated, and the results (when released) should be reliable.

4) Metallographic examination: Examination of the cerium metal, which was begun only recently, indicated that the impurities are concentrated in the grain boundaries.

5) Electrical conductivity: Precise measurements of the electrical conductivity of high-purity polycrystalline samples of the Reno cerium at temperatures near that of liquid helium have been undertaken to evaluate the relationship between this property and the purity of the metal. Matthiessen's rule[4] would indicate the possibility of such a relationship which has been demonstrated for gold[5] and copper, silver, and aluminum[6]. In general, this method has proved sensitive only to impurities that exist as solid solutions.

Discussion

Only a few of the basic factors involved in electrowinning rare-earth metals are discussed herein. Obviously, much remains to be completed before this laboratory procedure can be turned into an economically feasible process.

A) **Apparatus:** The reactivity of rare-earth metals is well known. Moisture, air, etc., all react with cerium metal, so that it becomes necessary to conduct experiments either in helium or argon atmospheres. The degree of success in achieving such an inert atmosphere in large measure determines whether the metal produced is valuable.

B) **General Considerations:** A comparison of the electrowinning of cerium and aluminum metals discloses many similarities and some important differences.

1) In each instance the composition and purity of the carbonaceous anode are of primary importance. Any impurities in the anode eventually find their way into the metal produced. The removal of many of the impurities from the anode can usually be accomplished by graphitization. This procedure creates other difficulties because of the high thermal conductivity of graphite. The aluminum cell, sacrificing purity as required to keep the cell in operation, uses a carbon anode. When higher purity cerium is prepared, the cell arrangement may have to accommodate a high heat loss. One alternative to the use of dc heating solely would be a provision for internal ac heating. The use of such a setup might require care in avoiding any possible interference between the dc and ac electrical circuits in the cell.

2) In each instance the cell container is a deciding factor. The aluminum cell, with a carbonaceous bottom and sidewalls, employs a frozen bath layer on the walls in a modified *skull* technique that is very effective. The bottom of the cell, being the cathode lead, cannot be covered by solid bath but must be open to the molten aluminum. In the larger cerium cell, it may be possible to arrange a more complete skull, with or without internal ac heating, to prevent reaction of the cerium metal with the container. The amount of impurities, both metallic and nonmetallic, introduced into the metal by contact with carbon or graphite is a matter of continuing research.

3) Both electrowinning cells use fluoride-base molten baths and feed oxides into the melt. In each cell the need for continuous oxide feed-mechanisms is self-evident.

4) One significant difference between aluminum and cerium is that the aluminum cell is operated essentially open to air.

5) A major difference is the fact that cerium may exist in the molten bath in both tri- and quadrivalent form, but virtually all the aluminum is trivalent except a small amount that is monovalent. This statement does not mean that trivalent or quadrivalent ions, as such, actually exist, since they probably would be in the form of complex ions.

C) **Oxygen Gas Found in the Cell Atmosphere:** The experiments here outlined seem to preclude an air leak into the controlled atmosphere chamber as the source of oxygen found after electrolysis is started in the cell. Absorption, adsorption, and en-

trainment of air as the oxide is fed into the cell also seem improbable. One is forced to conclude that oxygen is formed as part of the electrolytic operation. This conclusion, while at first startling, is not unreasonable and was reached during operation of some aluminum reduction cells, particularly at anode effect[1]. Hence, the production of molecular oxygen may be a function of the anode current density and also a function of the quality of the carbon or graphite anode used in the electrolysis.

If one assumes that the oxygen is produced as part of the electrolysis, the next problem is to locate its source. Two possibilities appear most prominent: 1) electrolysis of residual water in the melt, or 2) electrolysis of an oxygen *donor* formed on addition of the feed oxide to the molten bath. At present, there is no evidence to allow differentiation between these possibilities. At any rate, the formation of oxygen at the electrode does not invalidate the method, the aluminum reduction cell having been operated for extensive periods of time with similar problems.

The importance of this molecular oxygen formation is easily understood. It attacks the hot carbonaceous anode and weakens it by reacting both below and above the melt line. It can alter the gaseous atmosphere around the anode and in so doing can cause interruption of the electrolytic current. It reacts with any metal that has lost its cathodic protection, causing a loss of product. Experiments are being conducted to allow evaluation of these possibilities.

D) **Valence of Cerium Reduced in the Electrowinning Experiments:** Two valence states of cerium in the cell bath complicate any identification of the actual ion forms in the molten salt bath and make calculation of current efficiencies difficult, if not impossible.

The possibility of electrolytically reducing the trivalent ion form in the melt opens new vistas. It may be possible to use cerium dioxide that has been partly or completely reduced to the trivalent form by a carbothermal reaction, or some other mechanism, in the cell before the electrolysis. An alternative is to feed the trivalent oxide directly into the cell bath. A third possibility is the use of trivalent feed but not in an oxide form. All of these possibilities remain unevaluated at the present time.

E) **Nodule Formation:** In any evaluation of a sample, the assumption must be made that it is representative of the whole. When the metal is in the form of many small nodules, this criterion cannot be met without remelting the nodules. Remelting probably would change the properties of the metal and produce results that could not then be related to its original state.

Nodule formation is characterized by lack of homogeneity (shown by chemical analysis) between samples in a single experimental run. The comparison between runs is even less likely to produce significant information about factors that are important in the experimental procedures.

The exact causes of formation of nodules rather than a single large ingot have not yet been determined. The trends outlined in the experimental section, however, afford a basis for speculation:

1) Intermittent feed probably results in changed bath properties, when the oxide is added. In particular, the surface tension of the melt would be expected to change as the oxide dissolved. If the melt then withdraws from its normal intimate contact with the anode, it would probably cause interruption of the electrolytic current, followed by a temperature rise at the anode surface. These effects have been noted in aluminum reduction cells[1]. Local saturation of the melt may also cause similar changes in its character, leaving undissolved solids floating on or suspended in the liquid.

2) If the metal comes into contact with carbonaceous material when not connected to the cathode, the reaction may change the surface character of the liquid metal, preventing agglomeration into a large ingot.

3) Interruption of the dc for any reason has several possible effects, all of which result in loss of product. The electrowinning procedure forces the cerium ion, whatever its state and form in the bath, to be reduced to metal. Removal of the cathodic protection from the metal in contact with the cathode permits spontaneous reversal of the reduction step, and the metal returns to the ion form in the melt. A reaction between the cerium metal and the bath also is expected when the metal drops away from the cathode.

The above discussion reemphasizes the need for further basic research to determine the physical properties of the materials as used in preparing higher purity reactive metals.

Summary

1) Apparatus for electrowinning higher purity ingot cerium and the equipment used for measurement and control of inert atmosphere have been described.

2) Oxygen gas has been identified in the argon and/or helium atmospheres used in the electrowinning chambers. Its source apparently has been traced to the electrolytic reaction itself.

3) The evidence presented indicated that quadrivalent ions alone could not be the species reduced electrolytically to cerium metal. Either trivalent ions alone, or a combination of tri- and quadrivalent ions, are reduced to the metal at the cathode in the cerium electrowinning cell.

4) Several possible reasons for the formation of small nodules rather than a large ingot of cerium were examined.

5) The present state of analysis of higher purity reactive metals was outlined.

6) The electrowinning procedures for aluminum were compared with those for cerium metal from fused fluoride-base melts.

References

[1] E. Morrice, C. Wyche, and R. G. Knickerbocker: Electrowinning of Cerium. Paper presented at the February 1959 meeting of the *AIME*, San Francisco, Calif.

[2] E. Morrice and R. G. Knickerbocker: Rare-Earth Electrolytic Metals. Paper presented at the November 1959 meeting of the ASM-AEC Symposium, Chicago, Ill.

[3] Journal of Metals (AIME): Editorial, vol. 11, (8), p. 363, June 1959.

[4] A. Matthiessen: *Annalen der Physik und Chemie*, Leipzig, vol. 7, 1864, pp. 761, 892.

[5] J. O. Linde: *Annalen der Physik*, vol. 10, 1931, p. 52; *Ibid*, vol. 14, 1932, p. 353; *Ibid*, vol. 15, 1932, p. 219.

[6] J. E. Kunzler and J. H. Wernick: *AIME Trans.*, vol. 212, pp. 856-860, Dec. 1958.

[7] V. M. Mashovets, M. F. Dogramadzhi, and E. M. Florinskaya: *Journal of Applied Chem.* (USSR), vol. 25, 1952, pp. 1023-1030.

ELECTROLYTIC COBALT IN KATANGA

This is another of the papers presented at the International Symposium on the Extractive Metallurgy of Copper, Nickel, and Cobalt, held during the 1960 Annual Meeting. Complete text and discussion of all papers presented at the five-session Symposium will be available at the end of the year as one of the continuing series of volumes published by Interscience Publishers, Inc., for The Metallurgical Society.

One of the world's most important cobalt producing regions is Katanga, where copper-cobalt ores, mined and concentrated by the Union Minière du Haut-Katanga, are treated for the recovery of these metals at the firm's Jadotville plant. This is a description of the processes employed, with particular emphasis on cobalt.

Summarized from a paper by M. A. Bouchat and J. J. Saquet

Mining rights of a large area of south-eastern Katanga have been granted to the Union Minière du Haut-Katanga. This area borders the Rhodesian Copperbelt and has large copper deposits.

Current mining operations deal with a mixed oxide-sulfide orebody with dolomitic gangue. Treatment follows the classical concentration pattern: sulfide flotation with ethyl xanthate, and oxide recovery with amyl xanthate after superficial sulfidization by sodium sulfydrate. The sulfide concentrates assay from 40 to 60 pct Cu, depending on the dissemination of the minerals; these concentrates contain about 2 pct Co, 30 pct Fe, and 13 pct S. Oxide concentrates vary from 15 to 22 pct Cu. The average Co:Cu ratio of all the concentrates runs from 5 to 10 parts of cobalt to 100 parts of copper.

Sulfide concentrates are sulfate roasted in a fluo-solid reactor to increase their solubility in a sulfuric acid solution (spent electrolyte) to about 97-98 pct for copper and 95-96 pct for cobalt.[1,2] The solubility of iron is relatively low.

Oxide concentrates are also submitted to a sulfuric-acid leach, but in these materials about 80 pct of the cobalt is in the trivalent form, which is insoluble in sulfuric acid solutions. Leaching is done by means of spent electrolyte from copper electrolysis which contains ferrous sulfate as a reducing agent for the trivalent cobalt ion. The conditions of the closed-circuit copper electrolysis are such that about 50 pct of the total iron sulfate of the spent electrolyte is in the divalent form. However, in some cases the amount of ferrous ion is not sufficient and powdered copper is then added to the leaching vessels to reduce the ferric ion to the ferrous form. In this manner leaching of 96-97 pct of the cobalt contained in the concentrates is ensured in all cases.

Fig. 1 shows the flow of materials in the copper circuit. Leaching takes place in six rows of four pachucas of 40 cu m capacity each; leaching time is 4 hr. After separation of the sandy part of the solids in Dorr classifiers, the solution flows by gravity to primary thickeners, followed by secondary thickeners and silica gravel filters. Sedimentation of the solids is favored by the addition of the flocculation agents known as Jaguar and Separan.[3]

The thickened pulps are washed by counter-current decantation, and the clear solution flows to the 480-cell copper electrolysis hall, the capacity of which is some 130,000 metric tons of copper per year.

Copper removal in cobalt circuit

A part of the overflow of the first washing thickeners, containing 40 gpl Cu and 20 gpl Co, is diluted and diverted to the cobalt circuit. The amount is determined by the cobalt equilibrium in the copper solution—in the long run a tonnage of cobalt equivalent to the tonnage dissolved—and the volumetric equilibrium of the copper circuit. This solution, together with recovered wash waters, makes up the feed of the cobalt circuit. The total volume is about 40,000 cu m per month.

A three-stage process (Fig. 2.) is used to strip copper from the solutions. The first step is a copper electrolysis which is separate from the main copper electrolysis and operated at a lower current density —15 amp per sq ft. Copper content of the solutions is reduced from 40 to 5 gpl. The partially decopperized solution is high in free sulfuric acid—70 gpl —and is used to leach copper-cobalt ores in a separate section.

This ore is coarsely ground in another section of the plant and fed to a row of four leaching pachucas as a pulp at a dilution of approx 1 liter of water to 1 kg of solids. Leaching is completed with about 15

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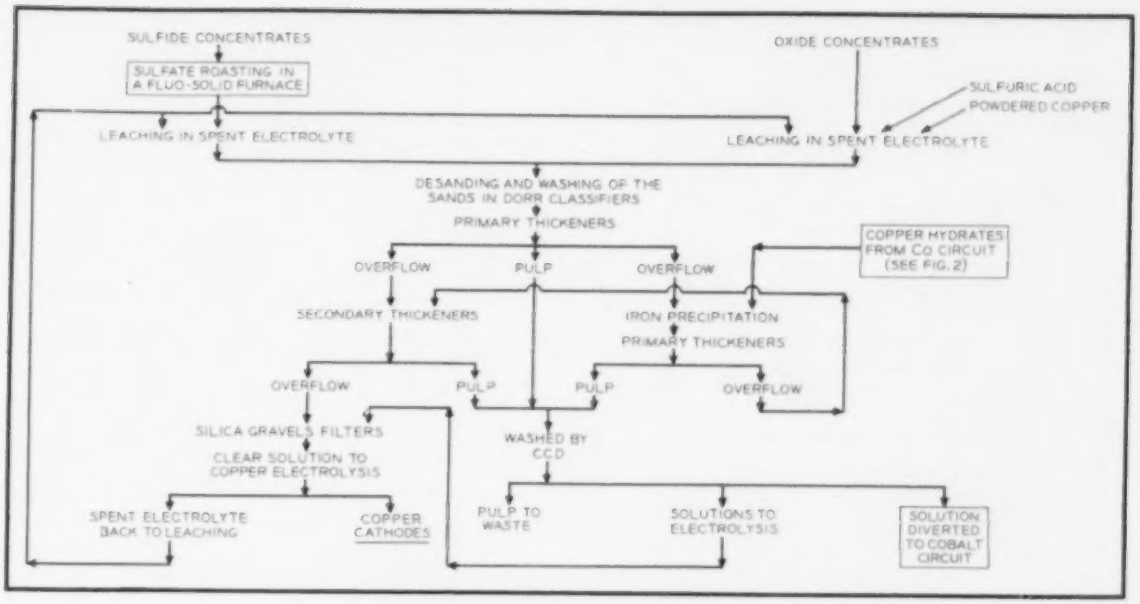


Fig. 1—Flow-sheet of copper circuit.

gpl of free acid in solution to ensure a good dissolution of both copper and cobalt in particles below 35 mesh. Most of the latter metal is also trivalent, and the process of its dissolution has been explained above.

After this leaching the pulp flows by gravity to a bowl classifier, where the coarse particles are separated and sent to the leaching pachucas of the copper circuit with stronger acidity. A complete extraction of copper and cobalt—even in sandy particles up to 35 mesh—is thus ensured.

The classifier overflow is separated into two parts. The first part flows to two 70-ft diam thickeners linked in series; flocculents are added to ensure a good clarification. The clear solution is sent back to the copper stripping electrolysis where ferrous iron is regenerated. This solution is recirculated to the leaching section, yielding more ferrous iron, which becomes available for the leaching of trivalent cobalt.

The second part of the classifier overflow, calculated to balance the volume of this closed-circuit second leaching electrolysis circuit, is mixed with the pulp underflow from the thickeners, mentioned above, and with a copper hydrate containing cobalt produced by a further step of the process (see Fig. 2). By adding a milk of lime to a controlled pH of about 3.3, most of the iron in solution is precipitated as ferric phosphate and ferric hydrate. The reaction takes place in a row of three 40-cu-m capacity pachucas.

The pulp is then sent to counter-current decantation washing; the pulp of the last thickener is sent to waste; and the overflow of the first thickener goes to another row of seven pachucas for further copper removal by the addition of lime. This addition is automatically regulated by means of a pH meter actuating a value situated on a pipe feeding the lime. Copper precipitation is limited in this step by setting the pH at a value of about 5.0. In this way excessive cobalt co-precipitation is avoided.

After the reaction, the pulp is thickened in two 70-ft thickeners, and—as said before—the underflow, after filtration on three 8x12 ft vacuum drum filters, is sent to the copper circuit for the precipitation of iron phosphate; the filtrate goes back to the thickeners mentioned above. After this step, the solution still contains 1 gpl of copper.

The operation also requires a second step of precipitation of copper by a milk of lime in two Dorr agitators of 80 cu m capacity each. The final pH is automatically regulated to a value of 6 to 6.2. During this step co-precipitation of appreciable amounts of cobalt takes place—about 1 lb of cobalt for 3 to 4 lb of copper. After thickening, the underflow, containing copper and cobalt hydrates, is mixed with the pulp from the leaching of the ore; in that way the free acid of the latter is beneficiated to dissolve the copper and cobalt hydrates of the former.

The overflow of the thickener, after acidification to a pH of 2.5 to accelerate the reaction, is passed through beds of calibrated cobalt granules where cementation of the copper takes place. After cementation, the copper content of the solution is reduced to 0.0010 gpl compared with 10 gpl of cobalt.

Cobalt precipitation

The purified solution is too dilute in cobalt to be fed to a closed electrolysis circuit; so, the cobalt sulfate is precipitated with a milk of lime at an automatically regulated pH value of 8.3. The reaction is carried out in three 12x7-ft Dorr agitators, and the precipitate is thickened in three 70-ft diam thickeners. The overflow, which contains 5 to 6 gpl magnesia and 0.4 gpl manganese, both in the form of sulfates, goes to waste. With the cobalt are precipitated the zinc and nickel in the solution and about 50 pct of the manganese.

The thickened pulp is a mixture of metallic hydrates and calcium sulfate. It is still very dilute—only 100 to 150 g of solids per liter of solution—and the solids must be filtered out in order to discard as much water as possible before introduction into the closed cobalt electrolysis circuit. This filtration takes place on four eight-disc Dorr Oliver filters. A so-called *blue cake*, about 10 mm thick, is obtained. It has a high water content: only 1 part of solid for 2

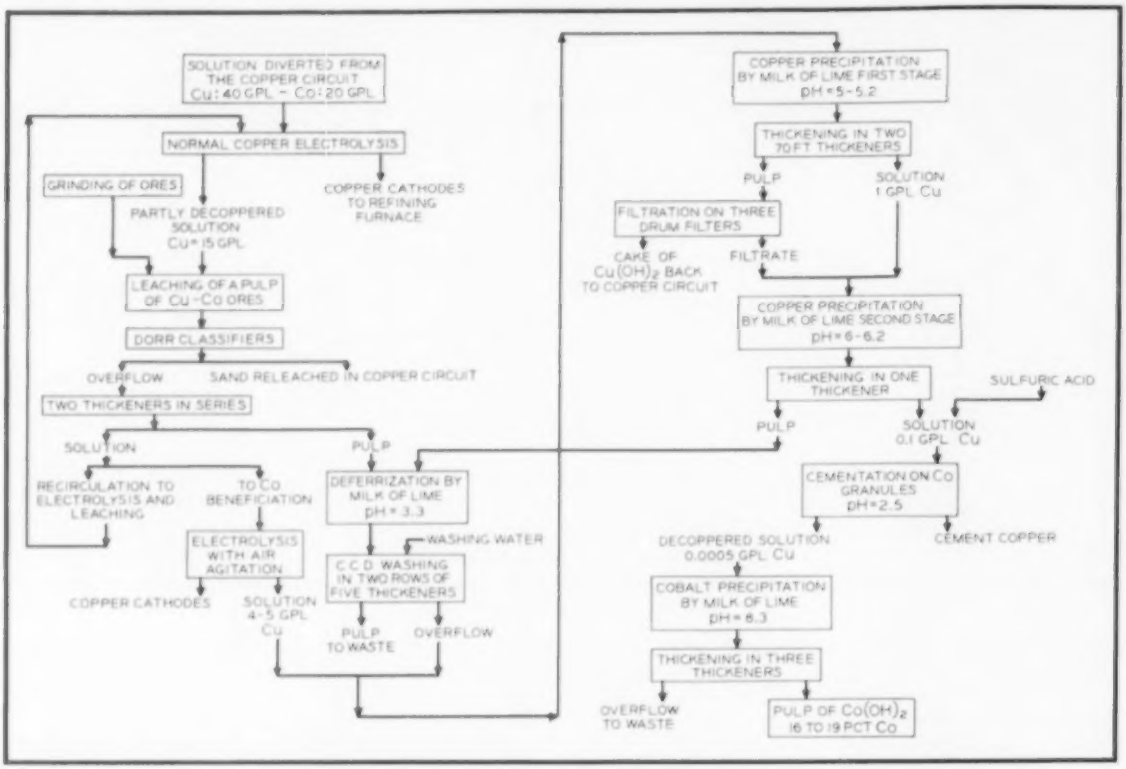

SOLUTION DIVERTED FROM THE COPPER CIRCUIT Cu: 40 GPL – Co: 20 GPL
NORMAL COPPER ELECTROLYSIS
GRINDING OF ORES
PARTLY DECOPPERED SOLUTION Cu = 15 GPL
COPPER CATHODES TO REFINING FURNACE
LEACHING OF A PULP OF Cu-Co ORES
DORR CLASSIFIERS
OVERFLOW
SAND RELEACHED IN COPPER CIRCUIT
TWO THICKENERS IN SERIES
SOLUTION
PULP
RECIRCULATION TO ELECTROLYSIS AND LEACHING
TO Co BENEFICIATION
DEFERRIZATION BY MILK OF LIME pH = 3.3
ELECTROLYSIS WITH AIR AGITATION
WASHING WATER
C.C.D. WASHING IN TWO ROWS OF FIVE THICKENERS
COPPER CATHODES
SOLUTION 4-5 GPL Cu
PULP TO WASTE
OVERFLOW
COPPER PRECIPITATION BY MILK OF LIME FIRST STAGE pH = 5-5.2
THICKENING IN TWO 70 FT THICKENERS
PULP
SOLUTION 1 GPL Cu
FILTRATION ON THREE DRUM FILTERS
CAKE OF $Cu(OH)_2$ BACK TO COPPER CIRCUIT
FILTRATE
COPPER PRECIPITATION BY MILK OF LIME SECOND STAGE pH = 6-6.2
THICKENING IN ONE THICKENER
SULFURIC ACID
PULP
SOLUTION 0.1 GPL Cu
CEMENTATION ON Co GRANULES pH = 2.5
DECOPPERED SOLUTION 0.0005 GPL Cu
CEMENT COPPER
COBALT PRECIPITATION BY MILK OF LIME pH = 8.3
THICKENING IN THREE THICKENERS
OVERFLOW TO WASTE
PULP OF $Co(OH)_2$ 16 TO 19 PCT Co

Fig. 2—Flow-sheet of de-ferrisation and de-coppering sections.

parts of water by weight. Since the solids average 18 pct Co, about 11 cu m of water per metric ton of cobalt is introduced with the cake. Consequently, the volumetric equilibrium of the cobalt electrolysis circuit is out of balance.

Up to now, the excess of solution is precipitated by milk of lime. But this creates a supplementary consumption of reagents: milk of lime for the re-precipitation of cobalt and sulfuric acid for the further redissolution of this cobalt hydrate. Another more economical process will be started soon in order to absorb this excess of volume: the blue cake will be filtered on vacuum drum filters instead of disc filters, and the water retained on the cake will be displaced by spent electrolyte sprayed on the surface of the cake. In that way the clear water can be expelled from the cake and join the wasted filtrate. Moreover, the amount of water introduced into the cake can be regulated by varying the amount of solution fed onto the cake. However, the intended displacement of the water is not ideal, and in practice, this secondary filtrate will contain a certain amount of solution. Hence, it must be sent back to the cobalt precipitation section, but this new technique will reduce by 80 pct the amount of cobalt re-precipitated and reagents consumed.

Cobalt electrolysis

Fig. 3 shows the flow of materials in the cobalt circuit. Blue cake is repulped by means of spent electrolyte which has a pH value of 3.0. Sulfuric acid must be added in proportion to the excess of lime and the presence of non-reactive magnesia in the cake.

The quantities of cake, electrolyte, and acid are proportioned in order to obtain only a partial dissolution of the cobalt hydrate. After this leaching, the solution is not clear, but carries a suspension of the calcium sulfate originally contained in the blue cake and a certain amount of undissolved cobalt hydrate. This pulp is fed into the cobalt electrolysis cells shown in Fig. 4: data on this unit is shown in Table I.

The electrolysis of such a pulp is made possible by blowing low-pressure air between the electrodes by means of a set of stainless-steel pipes.

During electrolysis, the deposition of cobalt on the blank is accompanied by the production of a corresponding amount of sulfuric acid which tends to acidify the bath. The respective values of the potentials of deposition of cobalt and hydrogen show that it is impossible to deposit cobalt electrolytically in acid solution with a good current efficiency. But, in the Union Minière process, solid cobalt hydrate stays in suspension. As soon as sulfuric acid is formed, it reacts with this hydrate, producing cobalt sulfate and water. This stabilizes the pH of the electrolyte at a value of 6.0 to 6.3 and maintains the amount of cobalt in solution unchanged during the electrolysis.

In such conditions, the metal forms some needles and nodules. To prevent short circuits, the cathodes must be lifted twice a shift and the needles scraped off by means of wooden sticks (Fig. 5).

After four days of deposition, the cobalt remaining on the blank must be stripped by hammering (Fig. 6). In practice, the production is divided into three parts of nearly equivalent weight: the metal stripped from the blank at the end of the cycle; the needles scraped during the cycle of electrolysis; and small lumps of cobalt accumulated on the bot-

tom of the cells and mixed with calcium sulfate and manganese dioxide. The latter is first passed through a wet magnetic separator to remove the non-metallics.

The cobalt from the electrolytic operation has the following analysis: 93-95 pct Co, 0.01 to 0.02 pct Cu, 0.2 to 0.6 pct Mn, 0.8 to 1.5 pct Zn, 0.15 to 0.25 pct Ni, 0.12 to 0.2 pct S, 0.02 to 0.05 pct Pb, and 0.05 to 0.08 pct Fe. It is obvious that this product is far from being pure; therefore, it must be fire refined.

Impurities in cathodic cobalt

Manganese: About 50 pct of the manganese dissolved from the ores and concentrates is precipitated with cobalt at a pH of 8.3. This manganese is dissolved and accumulates in the solutions of the cobalt electrolysis circuit. In practice, in a circuit in equilibrium, there is 1 g of Mn to 5 g of Co.

This impurity is eliminated in three ways:

a) with the solution sent back to the cobalt precipitation step;

b) by the formation of a scale of manganese dioxide on the lead anodes; this phenomenon is beneficial, for the deficiency of manganese in solution causes an unwanted deposition of cobalt trivalent oxide on the anode surface;

c) by deposition on the cathode with the cobalt; it should be remembered that, in acid solutions of pH 2 and less, manganese is not deposited at the same time as the cobalt, even if the solution contains up to 20 gpl of Mn for 50 gpl of Co.

Zinc: All of the ores treated normally contain a certain amount of zinc—1 to 2 parts of Zn to 100 parts of Co. During all hydrometallurgical operations, zinc goes along with the cobalt and is deposited with the cobalt during electrolysis. Zinc is finally eliminated in the fire refining.

Nickel: All of the ores processed by Union Minière contain, not only cobalt, but also a certain amount of nickel. The ratio of Ni:Co is quite variable—from 0.3 pct Ni to Co in the ores of the western zone of the concession to 5.0 pct in the eastern zone. Until now, we have not attempted to produce cobalt absolutely free of nickel, but we have fixed a maximum content of nickel in cobalt metal at 0.25 pct.

The nickel removal process used is known as the *De Merre process* and is patented by the Société Generale Métallurgique de Hoboken. The practice was described recently in several papers.[4-7] Briefly, the process applies the preferential sulfidization of nickel. The reaction is carried out in contact with cobalt metal as a reducing agent, and elemental sulfur or sodium sulfydrate as sulfidizing agent.

During electrolysis, the potential of nickel deposition is a little higher than the potential of cobalt deposition. As a result, the production of a cobalt containing 0.25 pct Ni is obtained with a ratio of 1.00 pct Ni to Co in the solutions of the closed electrolysis circuit. These solutions are submitted to the De Merre process. In practice, we do not try to eliminate all of the nickel, but we strip only about 50 pct of this metal and vary the amount of the solutions submitted to the nickel removal process so as to maintain a ratio of 1 pct or less of Ni to Co in the solution going to electrolysis. The selectivity of the reaction varies with the ratio of Ni:Co in the solution before purification and with the ultimate amount of nickel after purification.

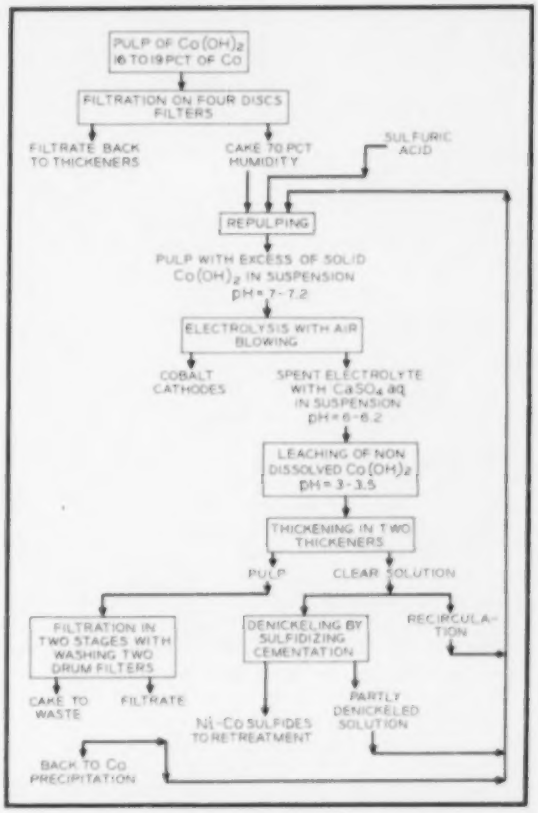

PULP OF $Co(OH)_2$ 16 TO 19 PCT OF Co
FILTRATION ON FOUR DISCS FILTERS
FILTRATE BACK TO THICKENERS
CAKE 70 PCT HUMIDITY
SULFURIC ACID
REPULPING
PULP WITH EXCESS OF SOLID $Co(OH)_2$ IN SUSPENSION pH = 7 - 7.2
ELECTROLYSIS WITH AIR BLOWING
COBALT CATHODES
SPENT ELECTROLYTE WITH $CaSO_4$ aq IN SUSPENSION pH = 6 - 6.2
LEACHING OF NON DISSOLVED $Co(OH)_2$ pH = 3 - 3.5
THICKENING IN TWO THICKENERS
PULP
CLEAR SOLUTION
FILTRATION IN TWO STAGES WITH WASHING TWO DRUM FILTERS
DENICKELING BY SULFIDIZING CEMENTATION
RECIRCULATION
CAKE TO WASTE
FILTRATE
Ni-Co SULFIDES TO RETREATMENT
PARTLY DENICKELED SOLUTION
BACK TO Co PRECIPITATION

Fig. 3—Flow-sheet of electrolysis and de-nickeling sections.

Table I. Electrolytic Cobalt Plant Data

No. of cells	80
Voltage per cell	5.0 v
Current density	40 amp/sq ft
Space between electrodes of the same polarity	80 mm
No. of anodes per cell (6 pct Pb, 1 pct Sb)	13
No. of cathodes per cell (mild steel)	12
Area of one anode	700x1000 mm
Area of one cathode	750x1030 mm
Current efficiency	85 - 88 pct

Characteristics of Electrolyte

Solids in suspension	40 gpl
pH inlet	7.0
pH outlet	6.0-6.3
Co	15-20 gpl
Mn	3 - 5 gpl
MgO	15 - 25 gpl
Ni	0.10 - 0.20 gpl
SiO_2	0.4 - 0.6 gpl
Saturated in $CaSO_4$	
Temp	50°C

Lead: The amount of lead varies with the degree of oxidation of the surface of the anodes. It is reduced by coating the anode surface with manganese dioxide, but a metal absolutely free from lead is never obtained.

Treatment of spent electrolyte

The spent electrolyte is a pulp containing about 40 gpl of solids. These solids are mainly $CaSO_4 \cdot 2H_2O$, originating from the cobalt precipitation by a milk of lime; but there is still a small amount of cobalt

hydrate left which prevents any pH drop during electrolysis.

This cobalt is dissolved by acidifying the pulp to a pH of 3.0. The pulp is then thickened; part of the clear overflow goes to the nickel removal section, and after elimination of the sulfides, all of the solution is again ready to repulp a new amount of blue cake and go to electrolysis.

The pulp from the thickeners is submitted to a double filtration on vacuum drum filters. Washing is done on the filter by means of clear water. A portion of the cake, which is essentially calcium sulfate with a small amount of manganese dioxide, is dumped to waste, and the remainder is sent to a cement plant for the manufacture of special sulfatized cements.

Pyrometallurgical refining

The main purpose of fire refining is the elimination of sulfur and zinc, but lead vanishes during the melt down, and manganese and phosphorus are oxidized and scorified; copper and nickel are practically unaffected. On the other hand, fire refining causes a pollution by iron and carbon from the electrodes, lining, and reagents. Finally because of the necessity of producing well-shaped and compact granules, a small quantity of silicon and aluminum must be added to the melt before granulation.

Melting furnaces: The plant is equipped with two Lectromelt CQ-type 2000-kva furnaces, each of 7000 lb theoretical capacity but in practice being overloaded to 12,000-13,000 lb of cathodes and return products. Before the final refining stage, a new lot of 1500 to 2000 lb of rejected or undersized granules is top-charged. The 8-in. diam electrodes are equipped with an automatic impedance regulation of an electro-mechanical type on one furnace, and of the Amplidyne type on the other furnace. The primary voltage is 6600 v, and the operator may select the secondary voltage within the range of 85 to 182 v between phases.

The whole furnace lining is basic. The bottom is made of rammed material, and the walls are made with magnesia brick which are low in iron—less than 0.50 pct Fe_2O_3. The roof is made of Austrian electromelted magnesia. Both the lining and the roof last about 80 to 100 melts.

Auxiliary Equipment: The melting and refining operations produce a large amount of fumes containing zinc, cadmium, and cobalt. These fumes are exhausted through a 0.6 sq ft slit situated between the roof and the wall, 45° away from the charging door on the opposite side of the column mast. An exhaust fan draws the fumes through a cyclone, a cooling vessel, and bag filters. The temperature of the fumes admitted to the bag filter is automatically regulated between 70° and 120°C by the quantity of water sprayed in the cooling vessel.

Operating procedure: The furnace charge is made up of the following materials: 5000 kg of cathodes, 500 to 800 kg of return scrap, and 700 to 1000 kg of discarded and undersize granules, making a total of 6200 to 6800 kg. The cathodes and scrap are charged in two parts. The first portion, weighing about 4 metric tons, is charged on top of a layer of 150 kg of lime. This part is charged by hopper bins which are supported by the crane so that the cathodes can slide by gravity into the furnace through the slagging door. The second part is charged manually.

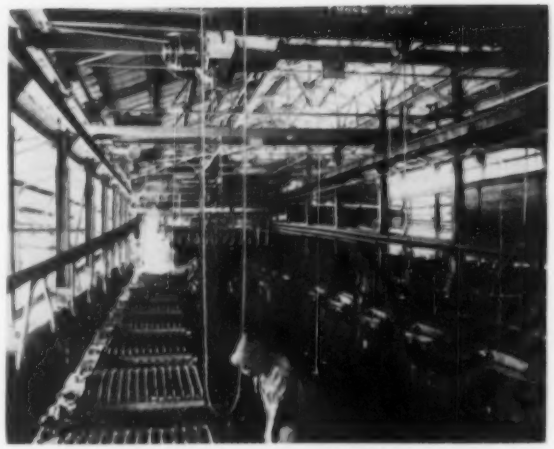


Fig. 4—Electrolytic cobalt tank house.

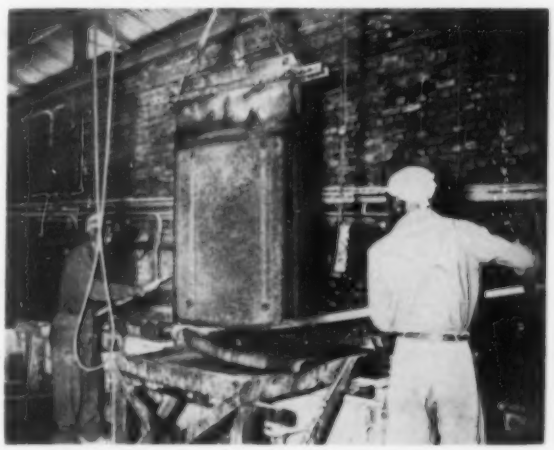


Fig. 5 (above)—Scraping cobalt needles off mild steel blank. Fig. 6 (below)—Stripping cobalt off blanks by hammering.

The total length of the melting period varies between 2½ and 3 hr. During this period the oxygen content of the cathodes stimulates the slagging of manganese and phosphorus in the lime. Lead is totally volatilized at the end of the melting period, as is about 80 pct of the zinc.

After the melt-down, the slag is skimmed off as well as possible, and further zinc removal proceeds. This is done with small poles, about 4 in. in diam (Fig. 7). By repeated poling operations, each of about 15 min duration, followed by 15 min of heating, the zinc content of the bath is lowered to a

figure of less than 0.01 pct. The number of poling operations varies between two and seven, but generally there are three or four, taking between 1 and 3 hr.

It is during the melting and poling periods that fumes containing zinc, cadmium, and cobalt are produced and recovered in the bag filters, described earlier.

After several operations, the slag should be skimmed off. Then a short de-oxidation with silico-calcium (1 kg) is required, and the bath is ready for desulfurization. This operation takes place in two or three steps. A slag is first built up with about 200 kg of lime mixed with 40 to 50 kg of fluorspar, and about 15 kg of quartz as fluidizing reagents. When the first slag is fluid, pulverized calcium carbide is blown into the bath through a wooden pipe fixed to a water-cooled steel pipe. The blowing cools the bath so that the slag has to be fluidized again by warming-up before skimming. The deslagging must be done as soon and as completely as possible to prevent any chance of pollution. This first desulfurization eliminates 80 to 85 pct of the sulfur. A second slag is made up, and when it is fluid, pulverized silico-calcium (33 pct Ca) is blown into the bath. Again, the deslagging must be done in the quickest way. Normally, these two desulfurizations last about 1 hr and draw 96 to 98 pct of the sulfur from the bath. The aspect of forged samples gives a good idea of the quality of the metal. If this test doesn't show good results, a third desulfurization with silico-calcium is necessary.

Owing to the desulfurization, the cobalt is polluted by carbon and silicon. To eliminate these impurities, the bath must be oxidized. At this point 700 to 1000 kg of rejected and undersize granules are top charged, and their remelting causes a slight oxidation of the bath. Oxidation is completed with oxygen blown through a forged cobalt five-hole tuyere.

The progress of the oxidation is controlled by checking the *set* of a small cobalt button. At the end of the oxidation, the carbon content is lower than 0.01 pct, and the silicon is as low as 0.003 pct. Unfortunately, the cobalt contains too much oxygen to be poured properly, and it must be reduced by the addition of 0.6 to 1 kg of silico-calcium-magnesium alloy. In order to avoid production of hollow granules, some aluminum must also be added. These metals are added after warming the bath up to 1620°C just before pouring.

The fire refining cycle is thus as follows:

Charging and melting	2½-3 hr
Poling	1-3 hr
Desulfurization	1 hr
Remelting of scrap	½ hr
Reduction	¼-1½ hr
Warming up before pouring	½ hr
Pouring	½ hr
Deslagging periods	1½-1¾ hr
Total Time Required	8-11 hr

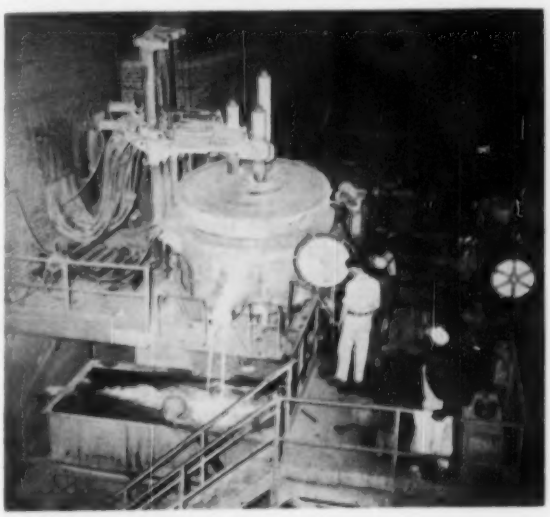
A power consumption of about 1400 kw per ton of products is required, and the consumption of electrodes is from 20 to 25 kg per ton.

Two conditions are needed to refine successfully: the melting must be completed before any refining stage can be started, and the bath must be maintained as clear as possible to avoid any pollution after refining. This requires many deslagging periods. Deslagging is done as often a possible with wooden pieces. Rakes must be used if the slag is too heavy; they are cast from cobalt to avoid the introduction of iron into the bath.

The slags are crushed and then treated on a magnetic separator. The magnetic part forms the return scrap to be charged with the cathodes. The non-magnetic part, mainly lime with 10 to 18 pct Co, is sent to electric reduction furnaces where it is mixed with a copper-cobalt sinter to produce a cobalt-iron-copper alloy known as white alloy. In these reduction furnaces, the cobalt from the bag filter fumes is recovered with the loss of the contained zinc and cadmium.

Fig. 7 (above)—Poling the molten bath by means of wooden sticks.

Fig. 8 (below)—Pouring refined cobalt into granulation tank.



The refined cobalt is granulated in a tank, which can be moved on a track and which contains 15 cu m of water. A fresh water supply of about 70 cu m per hr is fed near the bottom of the tank, and a circulation pump takes in another 30 cu m per hr at about 50 cm from the bottom and spreads it out on the upper water level of the tank as can be seen from Fig. 8. It is into this water stream that the cobalt is poured. Moving the tank along the track prevents the metallic granules from heaping up below the pouring spout.

The granules are dried in a rotary furnace, 3.1 m in length and with a diam of 0.90 m. This furnace is lined with fireclay bricks and is oil fired. The granules leave this furnace at about 200°C.

They are then polished in two rotating steel drums without any lining and with a capacity of about 1 ton. To evacuate the oxide dust formed during polishing, the two ends of the drums are provided with 10-mm holes, connected to a fan. Dust evacuation is improved by blowing dry air through the hollow shaft of the drum.

After polishing, the granules fall onto a double-deck Gyrex screen, yielding a +6 mm −6 mm +6 mesh, and an undersize fraction. The latter is returned to the furnace. The first two fractions leave the screen and drop onto a conveyor belt, the end pully of which is combined with a magnetic separator; all particles other than cobalt are discarded at this point. Along the belt conveying the +6 mm granules, a crew of four men pick out all badly shaped, hollow, or other undesirable granules. Finally, the granules are weighed on a semi-automatic scale and put in 500-lb drums, ready for shipment.

Future projects

At Luilu in Katanga, Union Minière is building a new plant for the beneficiation of oxidized and sulfide copper-cobalt concentrates. Generally speaking, the flow-sheet of the leaching operations is similar to that of the Jadotville plant, just described.

Changes mainly reside in the choice of modern equipment and especially in the introduction of a centralized process control. All chemical reactions will be automatically regulated by means of pH meters, densimeters, and flow meters; all of the indications concerning the different sections of the plant will be reported to a central dispatching room. From this central room, an operator will modify the set points of the regulators, stop and start the machines, pumps, and valves. This new plant, scheduled to start operations late in 1960, has been described in another paper.[8]

As far as cobalt is concerned, electrolysis will be performed according to new ideas. The goal is to produce marketable cathodic cobalt without any subsequent fire refining. Therefore, the cobalt solution, after having been submitted to the first two steps of copper elimination with a milk of lime, is to be fed to a combined nickel-copper stripping step —the *De Merre* process using sodium sulfydrate as a reagent. Thus, in this plant, the whole solution will undergo nickel elimination before being fed to the electrolysis. Copper elimination is always complete, but the degree of nickel elimination can be varied according to the desired purity of the final product—0.10 to 0.25 pct Ni.

Thereafter, a precipitation of zinc by hydrogen sulfide will be carried out, the pH during the reaction being maintained at 4.5 to 5.0 by adding calcium carbonate as a buffer. In that way, we will lower the zinc in solution from 300 to 1-2 mg per liter for a cobalt content of 20 gpl, and the co-precipitation of cobalt will be very low.

The following step will be a cobalt precipitation by milk of lime, and, after thickening and filtration, a leaching of the blue cake at a pH of 6.5. This high pH has been chosen owing to the propitious incidence of an absorption phenomenon which precludes the dissolution of some traces of zinc that may have escaped the operation of zinc precipitation. This phenomenon also precludes the dissolution of some zinc contained in the lime used for the cobalt precipitation. These traces of zinc and the zinc from the lime are returned with the leaching residues before the zinc precipitation step.

The leached pulp will be thickened and filtered, and the clear filtrate will be the feed of the electrolysis. Because of the acidification of the solution during electrolysis, a high circulation rate will be needed. But at a temperature of 60°C, when starting with a 50 gpl Co solution, a current efficiency of 75 pct with a deposition of 5 g of cobalt per liter of solution will still be obtained. The pH in the electrolysis cell is to be about 1.5, and under these conditions, there will be no manganese deposition on the cathode. The manganese equilibrium in this circuit will be regulated by deposition of manganese dioxide on the anode. This deposition has a beneficial effect on the lead content of the cathodic deposit.

According to laboratory test, the intended metal should have the following composition: 99.80 pct Co, 0.10 pct Ni, 0.01 pct Zn, 0.015 pct C, 50 ppm Pb, 15 ppm Fe, 50 ppm S, 10 ppm Mn, and 50 ppm Cu.

Conclusions

As can be seen, the hydro- and pyrometallurgy of cobalt at Union Minière have made appreciable progress during the last few years. The production of electrolytic cobalt started in 1944. However, until now the cathodes, produced in a pulp electrolysis process, have undergone fire refining.

The building of a second plant was started in 1958, and its cobalt production is being set in motion during the course of the second part of 1960. In this new plant, the process will comprise an electrolysis of clear, purified solution resulting in directly marketable cathodes.

The known amount of cobalt in the Katanga ores and the capacities of the existing and future plants of the company are such that Union Minière possesses incomparable potentialities for producing large tonnages of electrolytic cobalt of high quality.

References

[1] L. F. Theys and L. V. Lee: Sulfate Roasting Copper-Cobalt Sulfide Concentrates, JOURNAL OF METALS, Feb. 1958.

[2] L. F. Theys: Progress Report on Roasting Copper-Cobalt Concentrates, JOURNAL OF METALS, July 1958.

[3] F. J. Thoumsin and J. Capon: Emploi de floculants dans l'hydrometallurgie du Cuivre et du Cobalt, Compte-rendu du XXXIe *Congrès de Chimie Industrielle*, Liege, Belgium, Sept. 1958.

[4] B. Tourgarinoff, Dénickelage du Cobalt par cémentation sulfurante, Compte-rendu du XXVIIe *Congrès de Chimie Industrielle*, Bruxelles, 1954.

[5] B. Tougarinoff, Leonard, and Willekens, Dénickelage des solutions cobaltifères par cémentation sulfurante. Compte-rendu du XXXIe *Congrès de Chimie Industrielle*, Liege, Belgium, Sept. 1958.

[6] F. J. Thoumsin and M. Bouchat: Opération continue de sulfuration pour la séparation du cuivre et du nickel des solutions cobaltifères, Compte-rendu du XXXIe *Congrès de Chimie Industrielle*, Liège, Belgium, Sept. 1958.

[7] R. Breckpot: Sur le mécanisme de la séparation hydrométallurgique du nickel et du cobalt par réduction dépolarisée. Compte-rendu du XXXIe *Congrès de Chimie Industrielle*, Liège, Belgium, Sept. 1958.

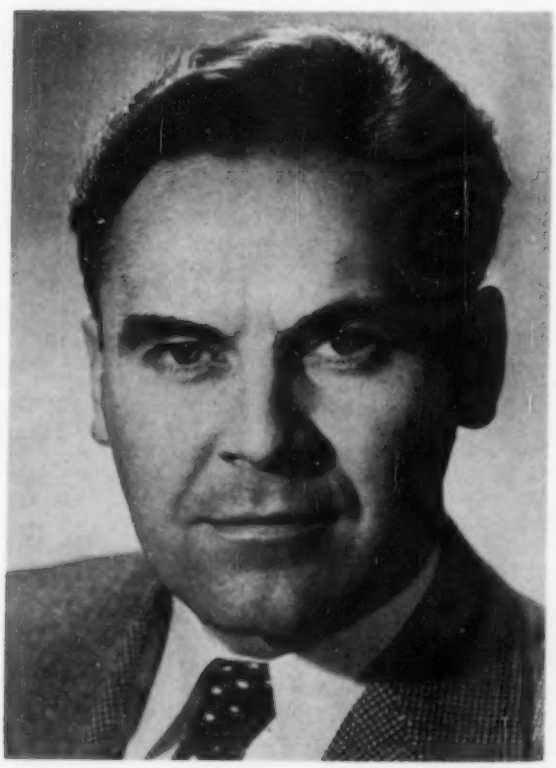
[8] Ch. Piedboeuf and F. Suys: Introduction du process control et de l'automation dans une nouvelle usine hydrométallurgique du Katanga, Compte-rendu du XXXIe *Congrès de Chimie Industrielle*, Liège, Belgium, Sept. 1958.

STEPS TOWARD ENGINEERING UNITY

The last century has witnessed the organization of professional societies in all fields of science and engineering. The consolidation of these societies into a unity such as the American Medical Association has been the aim of leaders in technology for many years. The following paper summarizes the actions taken and the plans proposed toward this much needed goal.

by Will Mitchell, Jr.

The AIME Board at its February, 1960, meeting appointed an ad hoc committee to study Inter-Engineering Society Cooperation. There are in this country twenty to thirty or perhaps more technical societies for engineers and engineering scientists. Most of these organizations are autonomous and there is little or no cooperation among them. Many of the societies do not touch the lives of our Institute members. However, the several societies that are closely associated with the AIME have, at various times, banded together in cooperative organizations to handle specific situations. It is felt by many engineers that the functions of these various cooperative organizations should be consolidated, possibly along lines similar to the American Medical Association and the American Bar Association. The AIME committee for Inter-Engineering Society Cooperation was set up as an information gathering group, to report its findings to the Board. The following is a progress report of the investigations carried on to date.



WILL MITCHELL, JR., is the chairman of the Inter-Engineering Society Cooperation Committee of the AIME.

The committee was comprised of Robert M. Mahoney of the Metallurgical Society, Douglas Ragland of the Society of Petroleum Engineers, and Will Mitchell, Jr., of the Society of Mining Engineers. The committee met in New York in February and and March, 1960 and again in May in Chicago. The preliminary study has indicated that the following organizations enter into the professional lives of our members: The five Founder Societies (AIME, ASCE, ASME, AIEE, AICHE), United Engineering Trustees (UET), Engineers Council for Professional Development (ECPD), Engineers Joint Council (EJC), National Society of Professional Engineers (NSPE), and National Council of State Boards of Engineering Examiners (NCSBEE).

Each committee man was assigned one or two of the groups to study. Mahoney covered the activities of the EJC and the NCSBEE; Ragland covered the activities of the ECPD; and Mitchell covered the activities of the NSPE. Some of these organizations are federated groups such as EJC and ECPD; others are direct individual member participation groups, such as the Founder Societies and NSPE. A brief history of these organizations follows. The purpose of each group and the nature of its membership is described.

The Five Founder Societies

The term *Founder Society* is a convenient term used in grouping several early technical societies. Included in the group are the American Society of Civil Engineers founded in 1852, the American Institute of Mining, Metallurgical, and Petroleum Engineers founded in 1871, the American Society of Mechanical Engineers founded in 1880, the American Institute of Electrical Engineers founded in 1884, and the American Institute of Chemical En-

gineers founded in 1908. These five societies assumed the responsibility for forming and administering the United Engineering Trustees, and have been the societies most active in the fund raising campaign for the new engineering building at the United Nations Plaza. The Founder Societies also provided the initiative in organizing several other cooperative professional and technical efforts.

United Engineering Trustees, Inc.

In 1904, Andrew Carnegie donated $1,050,000 to the engineering societies for a building to house them and their activities. During that year, representatives of the American Institute of Mining Engineers, the American Society of Mechanical Engineers, and the American Institute of Electrical Engineers met in New York and created The United Engineering Society (UES), as a joint holding corporation, to administer the proposed new home. The building in New York was completed by 1906 and UES assumed management. Prior to this time and during the time of negotiation the American Society of Civil Engineers was housed in its own quarters in New York. However, in 1916, ASCE decided to join with the other three Societies and in so doing became the fourth Founder Society to occupy the Engineering Societies building. United Engineering Societies became The Engineering Foundation, Inc., in 1930, and a year later it acquired its present title, United Engineering Trustees, Inc. In 1958 the American Institute of Chemical Engineers became the fifth Founder Society to join the UET corporation. Three trustees of each society are appointed by the respective boards of directors of the Founder Societies.

During recent years it was decided that the facilities of the original Engineering Societies building were no longer adequate space-wise or design-wise to administer the requirements of the mushrooming engineering activities. Subsequently, in 1958, a national fund raising campaign was inaugurated to provide new facilities for the profession. The success of the campaign is showing tangible results in the building program being carried on today. The accomplishments of UET over the past 56 years is a classic example of the ability of engineers of different disciplines to cooperate in a common cause.

Engineers Joint Council

In 1916 a Council of five representatives from each of the four Founder Societies and the UES was formed as a federation, "to provide for the convenient cooperation between Founder Societies, for the proper consideration of questions of general interest to engineers and to the public."

This group, called The Engineering Council, became in 1921 The American Engineering Council (AEC) with these defined objectives: 1) To further the public welfare wherever technical and engineering knowledge and experience were involved; 2) To consider and act upon matters of common concern to the engineering and allied technical profession. Herbert Hoover, an AIME member, then US Secretary of Commerce, was the first President of AEC. The AEC operated effectively until the depression of the thirties. By 1940, when drastic reductions in contributions impaired the activities of the Council, the membership had grown to include 54 national, state, and local societies. The AEC collapsed in 1940 when three of its largest contributors withdrew. The funds remaining were held in trust for a future similar organization. The dissolution of AEC demonstrated the inherent weakness of a federation type of unity organization.

In 1941, the Engineers Joint Council (EJC) was formed "to consider joint efforts in national defense". The Council was composed of the officers of the Founder Societies. It was at first called the *Joint Conference Committee*, then it became the *Engineers Joint Conference* and finally in 1945 it became the *Engineers Joint Council* at which time a constitution was adopted. In 1943 each member society provided three representatives to recommend a plan for the organization of the engineering profession. After considerable discussion and negotiation it was decided in 1952 to admit to EJC other Societies in addition to the five Founder Societies, and to provide for representation from the constituent societies proportionate to their membership. EJC Board membership is by appointment by the boards of directors of the technical societies. By 1958 EJC included in its federation 11 constituent societies, 3 associate societies, and 7 affiliate societies, and claimed a federated enrollment of "almost 300,000 individual members". The activities of the EJC include, the Engineering Manpower Commission. Panels on Atomic Energy and National Water Policy, and Committees on Air Pollution, Automation, Engineering Sciences, Honors, International Relations, Public Relations, and Recognition of Specialties in Engineering.

Engineers Council for Professional Development

In 1932 representatives of the five Founder Societies, the National Council of State Boards of Engineering Examiners, the Society for the Promotion of Engineering Education (since called American Society for Engineering Education), and the Engineering Institute of Canada, established the Engineers Council for Professional Development (ECPD). Originally the Council was formed "to advance the engineer professionally through the cooperative support of those national organizations directly representing the professional, scientific, educational, and legislative phases of the engineer's life". However, now ECPD concerns itself with: 1) Education and development of young engineers, 2) Establishment of standards for engineering education, 3)Accreditation of engineering curricula, and 4) Canon of ethics,

ECPD is a federation governed by a board made up of three representatives of each of the societies mentioned above. Representation is made by appointment by the boards of directors of the various societies involved. Through the years the splendid cooperation of the member societies and the wisdom of judgment of the various governing boards of ECPD have engendered a great respect from engineers and the public to the extent that ECPD is mentioned as an authoritative voice in the laws of 41 states in our country.

National Society of Professional Engineers

The National Society of Professional Engineers was formed in 1934. Ever increasing technological specialization led to the formation of more and

more technical societies, but it was realized that there were problems common to all engineers especially in their professional responsibilities. It was recognized that the advancement of the arts and sciences of engineering was receiving adequate attention by the various technical societies, hence NSPE proposed not to interfere in the technical field. It was devoted to the common interest areas of professional, social, economic, and ethical fields. NSPE is not a federation, but is similar in organization to the Founder Societies in that it is composed of individual dues-paying members who meet on the local, state, and national levels. In summary NSPE is: 1) Composed of professional engineers only, 2) Made up of individual dues paying engineers each having a voice in all affairs, 3) Operating at the three levels of interest of the citizen engineer—local, state, and national.

Registration laws covering the practice of engineering have been enacted in all states now; hence, registration has become a basic requirement for admission to membership in NSPE. A few exceptions to this basic requirement exist but these are beyond the scope of this report. There is only one grade of membership in NSPE at the present time.

National Council of State Boards of Engineering Examiners

Wyoming, in 1907, passed the first law requiring registration of engineers in our country. By 1920, eleven states had laws on their books covering registration of engineers. In that year, the National Council of State Boards of Engineering Examiners (NCSBEE) was founded "to promote the public welfare by improving professional engineering standards through efficient administration of State Engineering Registration Laws, by facilitating interstate registration of engineers, and by defining and maintaining national qualifications for registration". NCSBEE is actively working on a revised Model Law which the council hopes will be adopted by all state legislatures in order to provide uniformity and consistency among the states. Membership in the state boards is by appointment by the governor or an authorized representative of the state legislative body and these members are selected in general upon recommendation by the state or national professional societies, or both.

Recently NSPE canvassed its membership of over 50,000 with a questionnaire on *Organization of the Profession*. It is interesting to note that of the 15,000 replies received, 20 pct of the members of NSPE also belong to ASCE, 15 pct belong to AIEE, 13 pct to ASME, 2.7 pct to AIME, and 2.6 pct responding claim membership in AIChE. It was indicated that 75 pct of the members of NSPE belong to one or more technical societies. NSPE in its Policy No. 51 has recommended that every member of NSPE should "join and support the work of the technical society which serves his particular field of practice".

AIEE has proposed what is called the Functional Plan. The authors of the plan hope that it will be the first step toward unity in the profession. It attempts to delineate the scope of the responsibilities of EJC, ECPD, and NSPE by proposing that: 1) Cooperative action on technical and other related matters be a function of the Engineers Joint Council; 2) Cooperative action in the field of engineering education, and ethical practice be the responsibility of the Engineers Council for Professional Development; 3) The National Society of Professional Engineers be relied upon to further the development of the professional interests of the engineers. The ASME has gone on record as being in favor of the plan.

Another recent plan proposes the consolidation of the Engineers Council for Professional Development and the Engineers Joint Council and the creation of a third department to handle professional activities. Under this latter plan registration of members would probably not be a requirement.

Any realistic plan for unity, however, must take into consideration some tax regulations that have been established by the Internal Revenue Department. The department classes voluntary membership organizations into: 1) Educational, scientific, religious, and charitable (501-C-3). The Founder Societies, United Engineering Trustees, and Engineers Council for Professional Development fall under this classification. These organizations can receive donations and gifts that are tax deductible to the donor. They are also privileged to receive a real estate tax exemption. 2) Trade associations, chambers of commerce, etc. (501-C-6) whose objectives include the improvement of the economic welfare of the members. Organizations such as the American Mining Congress, the Mining and Metallurgical Society, the National Society of Professional Engineers, the American Medical Association, the American Bar Association, all fall in this group. These groups can not receive the tax benefits mentioned above. No funds from organizations listed under Group 1 above can be contributed to the organizations listed under Group 2. However, funds from the organization listed under Group 2 may be devoted to those listed under Group 1.

The Engineers Joint Council, before its recent incorporation, was classified under 501-C-6. Under the new charter all reference to professional-type activities has been omitted and EJC has requested reclassification as a 501-C-3 group.

This brief resume has been presented to refresh the background of the Inter-Engineering Society Committee and the AIME Board. For a more detailed study the reader is directed to the list of references. The committee has used these publications for this report and has quoted freely from them. No conclusions or recommendations are being made at this time.

References

The Organization of the Engineering Profession (NSPE).

Organizing the Engineering Profession to Achieve Unity (AIEE).

Organization of the Engineering Profession—Background and NSPE Member Opinion (NSPE).

EJC Annual Report, 1958.

Charter and Rules of Procedure, Engineers Council for Professional Development, revised Oct. 20, 1951.

The Registration Bulletin, published quarterly (NCSBEE).

STRESS CORROSION OF AUSTENITIC STAINLESS STEEL

by D. van Rooyen, Westinghouse Research Laboratories

Failure of austenitic stainless steels by transgranular stress corrosion cracking usually occurs in chloride or strong alkali solutions, under the influence of a tensile stress at elevated temperatures. Stress relief and strict control of the environment in which the steel is placed have been used to relieve the cracking problem, but these methods were not always possible or completely effective. It is known that cathodic protection can prevent the onset of cracking, and that it can even halt the propagation of an existing crack, but this remedy also has practical limitations. A fourth way of avoiding trouble is to select alloys of very high nickel contents, which have been found to resist stress corrosion cracking. It was usually assumed that minor changes in the composition of austenitic steels do not have much influence on cracking, so that little hope was held for a solution to the problem by a metallurgical approach.

More recently, however, the work of Uhlig and his co-workers[1, 2] on the effect of impurities has provided some hope that stress corrosion can be overcome by controlling the composition of the alloys. They showed that stable 20Cr-20Ni steels resist cracking in boiling magnesium chloride test solutions if their nitrogen content is kept low enough. While nitrogen had an adverse effect, they found that carbon could increase the resistance of these alloys to cracking. Silicon was classed as a beneficial addition to remelted 18-8 and 25-20 commercial steels, and cobalt and boron alloying additions were also thought to hold possibilities for improved stress-corrosion cracking resistance. This contribution to the knowledge of stress corrosion is of necessity still incomplete, but it has spurred several others to start work along the same lines in order to obtain a fuller understanding of compositional effects. It is hoped that their efforts in the metallurgical aspects of the problem will not only provide more data, but that they will lead to a clarification of the mechanism of cracking.

Other studies of the mechanism of cracking have been made, aimed at solving the problem through a thorough understanding of the basic processes involved. The approaches taken by different investigators have varied considerably. At present it is still impossible to decide which specific property of the alloys causes cracking to take place, if indeed there is only one process involved in the cracking mechanism. Recent work on the qualitative behavior of propagating stress corrosion cracks leaves doubt as to whether there are alternating steps of corrosion and instantaneous mechanical separation involved in the case of austenitic steels. Thus certain workers believe that brittle fracture accounts for the actual propagation of cracks and that corrosion only triggers this type of parting of the material. One interesting way in which this could happen has been postulated by Nielsen[3]. Essentially there is a build-up of solid corrosion products inside an

existing fissure, and since the corrosion products have a greater bulk than the metal from which they are formed, they will exert a considerable pressure on the walls of the crack. When the combined forces of the externally applied stress and the stress due to the precipitated metal compounds exceed a critical value, the crack will *jump* a short distance. This is followed by a standstill which is maintained until sufficient corrosion has taken place at the apex of the crack to provide enough corrosion products for the process to repeat itself. Another explanation of a similar overall mechanism has been proposed by Forty[4], who based his arguments on observations by Edeleanu[5] that cracking in brass is discontinuous. These arguments could equally be applied to austenitic steels: crack initiation is a chemical step, which is believed to set up an embrittled zone which cracks fairly readily under a tensile load, and in which the crack can reach a critical velocity. When such a crack reaches normal (non-embrittled) material, it can still propagate in a brittle manner into a normally ductile alloy, since its advance will depend on its velocity, and the rate at which metal can yield at its tip to relieve the stress. It is thought that alloys which are susceptible to stress corrosion cracking have a *low initial rate of yielding* (i.e., low dislocation velocity) so that crack propagation is supported.

Hoar[6] and his co-workers have made out a strong case for an entirely electrochemical mechanism of stress corrosion cracking of stainless steels. Their earlier work led to the conclusion that cracks could be formed by removal of metal along narrow planes by corrosion without the assistance of a brittle fracture step. Calculations, based on the rate of cracking, indicated that the anodic current densities required for the process would be of the order of ½ to 2 amps per sq cm. Under such conditions, however, one would normally expect considerable anodic polarization, and there was doubt whether such a rapid corrosion reaction could be self-supporting. More recently, however, they have shown that austenitic stainless steel samples can corrode at such high current densities with little or no polarization provided that there is no concentration polarization, and the metal is undergoing plastic deformation[6]. In contrast to this property of commercial stainless steel, nickel, which does not suffer chloride stress corrosion cracking, is found to polarize anodically under identical test conditions. In this connection, Basset and Edeleanu[7] found some indication that moving dislocations arriving at the surface of brass are more important from a corrosion point of view than stationary ones. They are also of the opinion that some stacking faults behave anomalously during corrosion, and tentatively suggest that the chemistry of reactions may be affected by the agglomeration of an alloying element around the stacking fault. The latter work was done with very thin foils, the structures and reactions of which were observed by direct-transmission electron microscopy.

The relationship of metal structure to stress corrosion cracking of the austenitic stainless steels is another aspect of the problem which has received considerable attention. However there is but little agreement even when comparing the more recent work. Uhlig[1], basing his suggestions on results provided by compositional studies, points out the possibility that interstitial nitrogen could help in the formation of crack-sensitive paths in the alloy during plastic deformation. This process could occur by either strain-induced precipitation of nitrides or incipient precipitates such as nitrogen atmospheres at dislocations. The diffusion of nitrogen is thought to be accelerated by strain and elevated temperatures. The nitrogen-rich areas are expected to be cathodic to the adjoining alloy where progressive corrosion and cracking could then occur. Other workers have attempted to ascertain whether there is any preferred plane or family of crystallographic planes along which cracking progresses. Earlier results indicated no such preference, but more recently Nielsen[8], and also Paxton and Reed[9] have obtained indications that some planes may be preferentially attacked. The latter workers found that in single crystals of 20-20 alloys cracks propagate along the cube plane which is more nearly normal to the applied stress. The crack zigzags from one to another cube plane when they lie approximately symmetrically to the direction of stress. However polycrystalline 18-8 and 20-12 alloys did not show the same preference. An interesting observation has been reported by Swann and Nutting[10], who found that there is an uneven distribution of solute in deformed alloys of low stacking-fault energy. Such alloys are also known for their susceptibility to stress corrosion cracking. Swann & Nutting suggest that segregation of solute to certain planes may lead to the formation of small cracks by an electrochemical mechanism. Along the same lines Uhlig[11] also proposed that cracking may be related to paths where defects predominate and where composition gradients exist. The over-all mechanism suggested by him, however, is one of conjoint electrochemical action and selective adsorption of ions which lower the energy of the crack walls. The idea of adsorption of ions which lower the surface energy and so explains the easy formation of cracks is not new, but there is little experimental data in the stainless steel field to support such a mechanism. It is to be hoped that some of the workers in stress corrosion will be able to overcome the great difficulty of carrying out work in this direction, and that they will provide some badly needed data.

A novel method of detecting localized sites of preferential attack has been followed by Gulbransen and Copan[12] who observe the shape of solid corrosion products of gas-metal reactions by direct transmission electron microscopy. They have shown that the oxide films on metals are mostly not uniform, but that whiskers and thin platelet shapes, perpendicular to the metal surface, are often formed in great abundance during high-temperature oxidation. Also, the composition of the atmosphere in which the oxidation is carried out has been shown to influence the type of oxide growth drastically. The influence of applied stress is also under study. In general their approach is that the local sites of *dry* reaction could be the same as those which will give rise to crack initiation in liquid media. Although, the corrosion products during aqueous corrosion will be soluble or may be precipitated at a distance from the place of reaction, the corrosion products in the gas-metal reactions are formed at the points or lines of activity, and may thus readily be studied. It will be a great step forward if these workers can show the correlation between the observed strange oxide shapes and defects such as dislocations within the metal structure. Of course, this will require the performance of extremely difficult experiments.

The above discussion has been based on the contents of only some of the more recent information which has become available. The report shows that, while opinions differ widely on some aspects of the problem, progress has been considerable and there seems to be reason for belief that the solution to the stress corrosion problem in stainless steels may not be too far distant.

References

[1] H. H. Uhlig and R. A. White: *ASM preprint* No. 167, 1959.
[2] H. H. Uhlig, R. A. White, and J. Lincoln: *Acta Metallurgica*, vol. 5, 1957, p. 473.
[3] N. A. Nielsen: Physical Metallurgy of Stress Corrosion Fracture (Interscience, N.Y.) 1959, p. 121.
[4] A. J. Forty: Ibid. p. 99.
[5] C. Edeleanu: Ibid. p. 79.
[6] T. P. Hoar and J. M. West: *Nature*, vol. 181, 1958, p. 835.
[7] G. A. Basset and C. Edeleanu: Physical Metallurgy of Stress Corrosion Fracture (Interscience, N.Y.) 1959, p. 117.
[8] N. A. Nielsen: Unpublished work.
[9] H. W. Paxton and R. E. Reed: To be published.
[10] P. R. Swann and J. Nutting: Structure and Properties of Thin Films (J. Wiley and Sons, Inc., N.Y.) 1959, p. 196.
[11] H. H. Uhlig: Physical Metallurgy of Stress Corrosion Fracture (Interscience, N.Y.) 1959, p. 1.
[12] E. A. Gulbransen and T. P. Copan: Ibid. p. 155.

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NEWS OF YOUR PROFESSION

Rocky Mountain Minerals Conference Drawing Near

The Rocky Mountain Minerals Conference, which will be held in Salt Lake City Oct. 5-7, will offer several sessions of particular interest to metallurgists. The conference will be held at the Newhouse Hotel, which will offer facilities for early registration the afternoon of Oct. 4.

The Welcoming Luncheon on Wednesday, Oct. 5, will have Maxwell E. Rich, Adj. General of the Utah National Guard as the featured speaker. Joseph L. Gillson, president of AIME, will be the guest speaker at the minerals luncheon.

The sessions on Thursday, which will interest metallurgists, will include papers on *Reagent Systems Design* by J. D. Vincent; *Electrolytic Tungsten and Molybdenum* by D. H. Baker, Jr., and J. B. Zadra; *Role of Micro-organisms in Leaching Processes;* and *The Vernal Phosphate Project of San Francisco Chemical Co.*, by D. L. King. The Thursday afternoon sessions will offer *The Silverton Project of Standard Metals Corp.*, by Edgar T. Hunter; and *Mining the Sea Floor* by L. E. Shaffer. Abstracts of all papers will be available by the time the conference opens.

An Exploration Symposium will be held on Friday morning, Oct. 7. It will cover ore search objectives; development of mines from ore search to production; costs of ore search; and ore search objectives, costs, and results in Canada. Speakers on the panel will include Francis Cameron, C. P. Pollock, and Charles C. Huston.

A final feature of the conference will be a trip to the Magna, Utah, plant of Hercules Powder Co. This is one of the world's most modern plants for research, development, and production of solid-fuel propellents and rocket engines. A dinner-dance will be held at the Newhouse Hotel on Friday evening.

Prof. N. F. Mott of Cambridge University Will Deliver IMD Lecture at Fall Meeting

Prof. Nevill Francis Mott of Cambridge University, England will deliver the Institute of Metals Lecture at the Fall Meeting of The Metallurgical Society on Oct. 18. His subject will be *The Work Hardening of Metal Crystals*. The Fall Meeting will be held in Philadelphia Oct. 17-20.

Prof. Mott has been professor of physics at the Cavandish Laboratory at Cambridge, since 1953, and Master of Gonville and Caium College, University of Cambridge, since 1959.

He was Henry Overton Wills professor, and director of the Henry Herbert Wills Physical Laboratories at the University of Bristol from 1948-1953.

Professor Mott has been a Fellow of the Royal Society since 1936. He received the Hughes Medal of the Royal Society in 1941, and the Royal Medal in 1953.

Educated at Clifton College, St. John's College, and Cambridge, Professor Mott has been a member of the Governing Board of the National Institute for Research in Nuclear Science since 1957. He was also president of the International Union of Physics from 1951-1957, and president of the Physical Society from 1956-1958.

His research work has been mainly on metals, semiconductors, and solid state physics. Professor Mott has written for various scientific periodicals about atomic physics, metals and semiconductors. In addition, he is the joint author of Mott and Jones' *Theory of the Properties of Metals and Alloys*, and other books in the field of solids and collision theory.

The aim of Professor Mott's lecture will be to summarize our present knowledge of the phenomenon of work hardening in terms of the behavior of dislocations. Recent work by transmission electron microscopy and the information deduced from it is contrasted with the information obtained from examination of slip lines. Although it is not felt that a complete understanding of the process is yet possible, some recent ideas are put forward which attempt to reconcile the available experimental evidence and to explain in outline the observed phenomena.

The practice of having a foreign scientist deliver the IMD Lecture on alternate years is made possible, in part, by a grant from the National Science Foundation.

In addition to the IMD Lecture, Professor Mott will speak at various universities and corporation research laboratories. He is scheduled to arrive in New York from England on Sept 27. He will spend the 28th at Bell Telephone Laboratories. Sept. 29-30 Professor Mott will be at the General Electric Laboratories in Schenectady. He will spend Oct. 3-5 at the Institute for the Study of Metals in Chicago. On Oct. 6-7, Professor Mott will visit Westinghouse and U.S. Steel in Pittsburgh. On Monday, Oct. 10, he will lecture at Yale University. He will spend the rest of the week at Massachusetts Institute of Technology. On Oct. 17-18, Professor Mott will be at The Fall Meeting of The Metallurgical Society in Philadelphia. He plans to return to England Oct. 19.

Iron & Steel Group To Meet in England

The forty-first meeting of the Iron and Steel Engineers Group will be held in Scunthorpe, England Oct. 24-27.

The program will include technical sessions at which papers on developments at Appleby-Frodingham, lighting in the iron and steel industry, failures in a blooming mill, and redeveloping an integrated iron and steelworks, will be presented and discussed.

There will also be visits to the works of the Appleby-Frodingham Steel Co., John Lysaght's Scunthorpe Works, Ltd., and Richard Thomas and Baldwins Ltd., by invitation of the companies. The Lincolnshire Ironmasters' Association will be the hosts at a dinner on Oct. 26.

Prof. Forward Named Scott Award Winner

Prof. Frank A. Forward, head of the Department of Metallurgy at the University of British Columbia, will receive one of the John Scott Awards of 1960 for his invention of the Forward Process for extracting nickel and other metals from ore concentrates. The award, consisting of a copper medallion and a cash prize of $1,000, will be presented Oct. 18 at the Fellowship Dinner of The Metallurgical Society during its Fall Meeting in Philadelphia. Among those attending will be the Canadian Consul General, H. A. Scott. The award was established in 1816 by John Scott, a chemist of Edinburgh, Scotland, who bequeathed $4,000 in trust to the city of Philadelphia. Today the fund has grown to $110,000.

The award will be presented by Donald C. Rubel, vice president of the Board of Directors of City Trusts.

Prof. Forward's process has substantially added to the world's supply of nickel. The extraction of this metal from several sizeable ore deposits, particularly those in Canada, was not commercially feasible until Prof. Forward invented his process which primarily consists of the extraction of nickel and other useful metals from ore concentrates by ammonia and water under pressure. The Forward process is not only less costly to operate on these ores, but also a substantially lower investment is necessary. Unlike older processes, nearby supplies of coal or oil are not required.

In the past, recipients of the award have included Marie Curie, for the discovery of radium; Orville Wright, inventor of the airplane, and Guglielmo Marconi, inventor of the wireless telegraph.

THE ENGINEERING INDEX

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AROUND THE SECTIONS

Bar & Shaped Products Conference Planned

A one-day conference on Bar and Shaped Products will be held at the Penn-Sheraton Hotel, Pittsburgh, on Jan. 18, 1961, under the sponsorship of the Mechanical Working Committee, Iron and Steel Division, The Metallurgical Society, and the Pittsburgh Section of AIME.

Papers will include discussions of the surface preparation and rolling of primary products and new developments in processing and testing.

NSF Program Offers Chance For Study In Scandinavia

Opportunities for a specialized scientific research and training for one-year period in Norway, Sweden, and Denmark are now available to five U. S. scientists under the terms of a $15,125 grant made by the National Science Foundation to the Scandinavian Council for Applied Research.

The foundation listed the following fields of science for which U. S. scientists may apply: acoustics, corrosion, chemical engineering, automation, microbiology, biotechnology, mass spectrometry, speech transmission, and calorimetry.

The foundation grant will provide on the average $825 for institutional costs, $1,500 for stipends, and $700 for travel for each U. S. participant. The balance of institutional costs and additional stipend will be provided by the Organization for European Economic Cooperation (OEEC), another sponsor of the program, called the *Growing Points* program. Studies can be started anytime of the year, in accordance with an agreement with the head of the *Growing Point* center.

Selection of U. S. participants will be made by the Scandinavian Council for Applied Research. Applicants should direct inquiries to the Scandinavian Council for Applied Research, Gaustadalleen 30, Blindern, Norway.

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29 West 39 Street,
New York 18, N. Y.

High Temperature Conference Set For Cleveland, April 26-27

The Cleveland section of AIME will sponsor a High Temperature Materials Conference on April 26-27, 1961 at the Hotel Pick-Carter in Cleveland. It will be held in cooperation with the IMD High Temperature Alloys Committee and Refractory Metals Committee. The presentation is expected to be patterned after the Cleveland meeting of 1957 which resulted in the publication of the book, *High Temperature Materials* edited by Hehemann and Ault, and published by John Wiley & Sons.

The subjects will cover materials suitable for use above 1600°F, as listed: 1) Requirements for High Temperature Materials, 2) Cobalt and Nickel Alloys, 3) Molybdenum and Columbium Alloys, 4) Tungsten and Tantalum Alloys, 5) Graphite and Refractory Compounds, 6) Coatings on Refractory Metals, 7) Processing and Fabrication of Refractory Metals.

The conference will be of interest to engineers and scientists working in the missile, aircraft, nuclear and related fields. Those desiring to submit papers on recent research and developments on the listed subjects, should send title and summary outlining the scope and detailed description of data by Dec. 1, 1960. These should be sent to:

Dr. H. P. Munger
Republic Steel Research Center
6801 Brecksville Road
Cleveland 31, Ohio

A detailed program will appear in a later issue of Journal of Metals.

Structural Materials Group Meets Oct. 18

The IMD Structural Materials Committee will meet at the National Metal Congress in Philadelphia Oct. 18. The meeting will be held in the Independence Room of the Sheraton Hotel at 8:30 a.m.

On Wednesday, Oct. 19, the committee will sponsor a symposium at the Fall Meeting. This symposium, which will be in the Pennsylvania East Room at 2 p.m., will have six speakers. The subject will be *Structural Materials in Cryogenic Design.*

EJC REPORTS ON TRIP TO RUSSIA

Engineers Joint Council delegation to the U.S.S.R.—Seated, L to R—Walter E. Lobo, consulting chemical engineer, New York, N. Y.; H. Russell Beatty, president of Wentworth Institute, Boston, Mass. Standing, L to R—Carlton S. Dargusch, advisor, Engineering Manpower Commission of EJC, of Columbus, Ohio; Oleg Hoeffding, economist, the Rand Corp., Santa Monica, Calif.; Sidney B. Ingram, director of technical employment, Bell Telephone Laboratories, Inc., Murray Hill, N. J.; and Robert M. Mahoney, manager, Industrial Relations Development, Union Carbide Corp., New York, N. Y.

In accordance with the Soviet-American program of scientific, technical, and cultural exchange, a group of six-men—representatives of the EJC—has recently completed a month of travel and observation in the USSR. The delegation was composed of men representing a cross-section of engineering specialties, engineering education, and economics. The members were: Walter E. Lobo, delegation chairman, consulting chemical engineer, New York, N. Y.; H. Russell Beatty, president of Wentworth Institute, Boston, Mass.; Carlton S. Dargusch, advisor, Engineering Manpower Commission of Engineers Joint Council, Columbus, Ohio; Oleg Hoeffding, economist, Rand Corp., Santa Monica, Calif.; Sydney B. Ingram, director of technical employment, Bell Telephone Laboratories, Inc., Murray Hill, N. J.; and Robert M. Mahoney, manager, industrial relations development, Union Carbide Corp., New York, N. Y.

Their foremost objective was to study the utilization of engineers and engineering technicians. The delegation visited 13 technical institutes, three technicums, and five enterprises—industrial plants—which were situated in Moscow, Leningrad, Kharkow, Rostov, Baku, Tashkent, and Smarkand.

Significant changes have been taking place in the Soviet education system. A goal has been set to admit no more than 20 pct of their high school students directly to higher education immediately upon graduation. The remainder of the graduates must join industry for two years before they may be admitted. The chosen 20 pct are not to be harbored in an academic tower. The curricula includes a year and four months of industrial activities. Intensive theoretical and mathematical studies combined with practical experience provide a high-quality engineering education. The delegation found that foreign languages and political economy were included in the course of studies to the exclusion of what we refer to as the humanities.

This program produces 108,000 graduate engineers each year. However, Mr. Lobo, spokesman for the group, stated that many engineers were assigned to jobs which would not be considered engineering posts in this country.

The Russian technicums represent a very effective four-year training program. The USSR Ministry of Higher and Secondary Education stated that 250,000 technicum students are graduated annually. This figure compares to the yearly graduation of 14,000 to 16,000 graduate technicians in the US. The delegation felt that not over 1000 of the above compare favorably with their Russian counterparts.

It is interesting to note that ap-

(Continued on page 817)

Price Will Increase For Transactions

Announcement has been made by The Metallurgical Society that effective January 1, 1961, the subscription price of the bi-monthly TRANSACTIONS OF THE METALLURGICAL SOCIETY OF AIME, will be increased for non-members from $20.00 per year to $30.00 per year. For non-members outside of the Western Hemisphere, a subscription will cost $32.00. These increases are necessitated by increased publication costs. There will be no increase of the yearly subscription to members, remaining at $5.00 per year.

Bound volumes, if ordered when paying annual dues will remain at $5.00 for members. If ordered later, the charge for bound volumes of TRANSACTIONS will be $6.50. Bound volumes for non-members will cost $35.00 after January 1, 1961.

Because of the expense of carrying bound volumes of back issues in inventory, the charge for back volumes will be increased to $35.00 for non-members on January 1, with 30 pct discount being given to members, or $24.50 per volume.

Members may wish to purchase copies of bound volumes now before the price increase becomes effective. The following price schedule for members will exist for the balance of the year for those volumes that remain in stock:

1958-1959
$5.00 per copy (non-members, $25.00)

1954-1957
$6.30 per copy (non-members, $9.00)

1953
$4.90 per copy (non-members, $7.00)

Metals Branch TRANSACTIONS for the years 1949-1952 inclusive are currently out of stock. Arrangements have been made with Johnson Reprint Corp. to reprint Volumes 1-4 of JOURNAL OF METALS, including TRANSACTIONS. A discount price will be available for members of AIME. This will make it possible for individuals and libraries to obtain a complete set of Metals TRANSACTIONS from 1949 to the present. Orders for the years 1949-1952 should be sent to Johnson Reprint Corp., 111 Fifth Avenue, New York 3, N. Y.

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MEMBERSHIP

Proposed for Membership
Metallurgical Society of AIME

Total AIME members on August 30, 1960, was 34,205; in addition 2,545 Student Members were enrolled.

ADMISSIONS COMMITTEE

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The Institute desires to extend its privileges to every person to whom it can be of service, but does not desire as members persons who are unqualified. Institute members are urged to review this list as soon as possible and immediately to inform the Secretary's office if names of people are found who are known to be unqualified for AIME membership.

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Doering, Harvey V.
Pennsylvania State University
Dolan, Robert W.
Rice University
Youngblood, James L.
Stanford University
Neiman, Alfred S.

NECROLOGY

Date Elected	Name	Date of Death
1955	Bachner, J. G.	Unknown
1948	Emrath, P. C.	July 4, 1960
1954	Fletcher, J. E.	Unknown
1907	Harrington, G. B. (Legion of Honor)	May 13, 1960
1940	Hett, Paul	July 31, 1960
1913	Hoffman, R. B.	June 1, 1960
1954	James, John C.	Unknown
1931	Jones, V. W.	Unknown
1960	Kapantais, T. P.	July 3, 1960
1956	Neely, F. H.	May 9, 1960
1917	Riddell, J. M.	July 13, 1960
1955	Swenson, G. R.	July 22, 1960
1922	Warner, George A.	June 26, 1960

Metallurgical Society Publishes Booklet

The Metallurgical Society has published *Careers In Metallurgy and Metallurgical Engineering*, a sixteen page booklet to aquaint students with the meaning of metallurgy and the possibilities for careers in the field.

The booklet was made possible by a $7500 grant from the National Science Foundation.

Illustrated with photographs and sketches, the booklet is divided into five chapters which answer the questions: 1) What is metallurgy and what is a metallurgical engineer; 2) What does he do; 3) What would such a profession offer me; 4) What does it take to be a metallurgical engineer; 5) How can I prepare to become a metallurgical engineer.

Copies of the booklet will be available to anyone desiring them. It will be distributed through the National Science Teachers Association.

The booklet was prepared by The Metallurgical Society's Education Committee, which is composed of A. W. Schlechten, Missouri School of Mines and Metallurgy; R. Schuhmann, Jr., Purdue University, R. W. Fountain, Union Carbide Metals Co.; W. O. Philbrook, Carnegie Institute of Technology; C. C. Long, St. Joseph Lead Co.; and J. J. Burke, JOURNAL OF METALS.

The inside back cover of the booklet contains a list of colleges and universities which have curricula in metallurgical engineering accredited by the Engineers Council for Professional Development (1959).

Education

(Continued from page 758)

in each of five fields, totaling 110 cash awards and corresponding certificates. Steel foundries will also receive certificates recognizing their cooperation in developing entries in behalf of users. Each entrant will receive a copy of the 1960 Steel Castings Handbook, a completely revised and rewritten volume of metallurgical, design, engineering, and production data.

There will be two classes of entrants: industry and student. Industry personnel, except steel foundry employees and SFSA staff members, are eligible to compete in the industry class and full-time students (including graduate students) are eligible for the student contest.

Entries for both classes must be made in one of five divisions: 1) end cost, 2) mechanical design, 3) metallurgical design, 4) redesign or conversion, and 5) unusual or unique.

Complete rules and format guides for the 1961 Centennial Product and Development Contest are available from the Steel Founders' Society of America, 606 Terminal Tower, Cleveland 13, Ohio.

EJC Reports . . .

(Continued from page 816)

proximately one third of the technical students were women. The women, it was believed, had been granted this opportunity because of a temporary shortage of men—due to World War II—rather than a desire for equal privileges for women.

This thought was supported by the fact that few women were observed to have reached top management positions. Management seems to stick to party lines—about 75 pct of the managers carry party cards.

A great stress is put upon the continuation of education. The higher social status which comes to engineers and scientists provides the impetus to take part in correspondence and refresher and upgrading courses. The members thought economic and social rewards spurred undergraduates to more serious endeavor than is noted in the average American engineering student. Approximately 75 to 80 pct of all students receive stipends for academic achievement.

Throughout their technical education the Russian students work in close cooperation with industry. As a consequence, they achieve a training in a specialty in addition to a broad basic education. On the other hand, most US students, bachelors candidates, do not receive any degree of specialized training until after graduation.

The US delegation concluded that we must become more serious in our view of education. Though the Russian system does not seem applicable to our country, much can be learned from it. According to the EJC group, the American student must be made to realize the importance of his training both to himself and to his country.

OBITUARIES

Dr. Harry K. Ihrig, vice president in charge of research for Allis-Chalmers Manufacturing Co., died suddenly Aug. 22. He was 62. Prior to joining the company in 1950, Dr. Ihrig had been associated for 16 years with Globe Steel Tubes Co., Milwaukee, as director of laboratories and vice president.

In 1947 he was awarded the Robert W. Hunt medal by AIME for the best paper published on iron and steel research.

Coming Events

Oct. 3, Chicago Section, NOHC, Fall Dinner Meeting, Phil Smidt's Restaurant, Hammond, Ind.

Oct. 5-7, Rocky Mt. Minerals Conference, Newhouse Hotel, Salt Lake City.

Oct. 12, Annual Meeting of the Cast Bronze Bearing Institute, Grove Park Inn, Asheville, N. C.

Oct. 12-13, Seventh National Symposium, American Vacuum Society, Cleveland-Sheraton Hotel, Cleveland.

Oct. 12-14, Fourth Conference on Analytical Chemistry in Nuclear Reactor Technology—First Conference on Chemistry in Nuclear Reactor Technology, Gatlinburg, Tenn.

Oct. 13-14, Southwestern Section, NOHC, Fall Meeting, Statler-Hilton Hotel, Dallas.

Oct. 13-15, 1960 Annual Meeting of the Non-Ferrous Founders' Society, Grove Park Inn, Asheville, N. C.

Oct. 17-18, Annual Convention, The Magnesium Association, Pick Carter Hotel, Cleveland.

Oct. 17-20, Fall Meeting of The Metallurgical Society of AIME, Sheraton Hotel, Philadelphia.

Oct. 17-21, Fall Meeting of the Societe Francaise de Metallurgie, Maison de la Chimie, Paris.

Oct. 23-26, Seminar on Rare Earth Research, Arrowhead Center, University of California, Los Angeles.

Oct. 28, Eastern Section, NOHC, 14th Annual Conference, Warwick Hotel, Philadelphia.

Nov. 4, Pittsburgh Section, AIME, 15th Annual Off-the-Record Meeting, Penn-Sheraton Hotel, Pittsburgh.

Nov. 8-11, First National Die Casting Exposition and Congress, Detroit Artillery Armory, Detroit.

Nov. 15, Buffalo Section, NOHC, 11th. Annual Fall Meeting, Statler-Hilton Hotel, Buffalo.

Nov. 14-16, Fifteenth Technical and Operating Conference, Steel Founders' Society of America, Hotel Carter, Cleveland.

Nov. 30-Dec. 2, AIME 18th Electric Furnace Conference, Morrison Hotel, Chicago.

Jan. 18, 1961, Conference on Bar and Shaped Products, sponsored by Mechanical Working Committee of The Metallurgical Society, Penn-Sheraton Hotel, Pittsburgh.

Feb. 26-March 2, 1961, AIME Annual Meeting, Chase & Park Plaza Hotels, St. Louis, Mo.

Mar. 13-14, 1961, 59th Annual Meeting, Steel Founders' Society of America, Drake Hotel, Chicago.

Apr. 10-12, 1961, 44th National Open Hearth Steel Conference and Blast Furnace, Coke Oven and Raw Materials Conference, Sheraton Hotel, Philadelphia.

Apr. 12-14, 1961, AIME International Symposium on Agglomeration, Sheraton Hotel, Philadelphia.

Apr. 26-27, 1961, Cleveland Section, The Metallurgical Society, Technical Conference on High Temperature Materials, Hotel Carter, Cleveland.

Oct. 23-26, 1961, Fall Meeting of The Metallurgical Society of AIME, Pick-Fort Shelby Hotel, Detroit.

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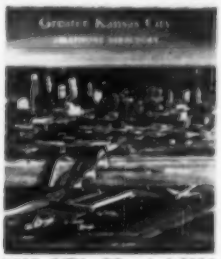

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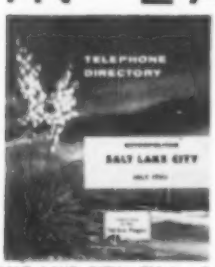

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